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UNITED NATIONS INDUSTRIAL DEVELOPMENT ORGANIZATION

INTERNATIONAL CENTRE FOR SCIENCE AND HIGH TECHNOLOGY
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SMR/543 - 2

EXPERIMENTAL WORKSHOP ON HIGH TEMPERATURE SUPERCONDUCTORS AND RELATED MATERIALS (BASIC ACTIVITIES)

(11 February - 1 March 1991)

" Cryostat Design " - PART II

presented by:

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Calibration of thermometers.

It is advisable to follow the instructions of the IPTS 68 * with regard to the establishment of fixed points, the checking of boiling points, and the testing of the purity of liquid refrigerants.
If calibration is to be supplemented by calibration at intermediate temperatures, it is best to use a combined platinum-type continuous-flow cryostat together with a secondary thermometer.

* C. R. Barber : Metrologia 5, 35 (1969)
R. A. Haefer : Cryopumping, Theory and Practice. Oxford, Clarendon Press 1989. p. 227 ff.

I

1968

TABLE A.4(a) Fixed point temperatures T_{68} according to the International Practical Temperature Scale IPTS 68, and the resistance ratio $R(T_{68})/R(273.15\ K)$ of a platinum resistance thermometer constructed in accordance with IPTS 68 [A12].

Fixed Point	T_{68} (K)	$R(T_{68})/R(273.15\ K)$
1. Triple point e-H_2	13.81	0.001 412 06
2. e-H_2 333.306 mbar	17.042	0.002 534 44
3. Boiling point e-H_2*	20.28	0.004 485 17
4. Boiling point Ne	27.102	0.012 212 72
5. Triple point O_2	54.361	0.091 972 52
6. Triple point Ar	83.798	0.216 057 05
7. Boiling point O_2	90.188	0.243 799 09
Ice point	273.15	1[t]
Triple point H_2O	273.16	—
8. Boiling point H_2O	373.15	1.392 596 68

*All boiling points at 1.013 25 bar = 101 325 Pa.

II

1976

TABLE A.4(b) Fixed point temperatures T_{76} according to the Provisional Temperature Scale EPT 76 [A13]

Fixed point	T_{76} (K)
Transition temperature of cadmium	0.519
Transition temperature of zinc	0.851
Transition temperature of aluminium	1.1796
Transition temperature of indium	3.4145
Boiling point of *He*	4.2221
Transition temperature of lead	7.1999
Triple point of e-H_2	13.8044
e-H_2 333.306 mbar	17.0373
Boiling point of e-H_2	20.2734
Triple point of Ne	24.5591
Boiling point of Ne	27.102

*All boiling points at 1.013 25 bar = 101 325 Pa.

III The International Temperature of 1990 (ITS 90)

- The thermodyn. (absolute) temp. is defined by the second law of thermodynamics.
- To establish the thermodyn. temp. scale, a fixed point is needed, which is defined by the triple point of water at $T = 273.16$ K. As this point lies at 0,01 °C, the conversion formula for the Kelvin & the Celsius scales is
$$T(K) = t(°C) + 273.15$$
- The absolute temp. scale is generally measured by the constant volume helium gas thermometer which is unsuitable for routine measurements.
- Hence, secondary thermometers are used, calibrated on the practical scales, which for their part coincide with the greatest accuracy with the thermodyn. scale

Secondary thermometers

TABLE 9.4. Thermometers for low temperatures

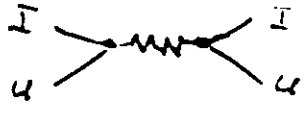
	Measurement range (K)	Sensor volume (cm^3)	Measurement power (μW)	Measurement accuracy (mK)	Reproducibility (mK)	Sources of supply
● Resistance thermometers						
Platinum, encased[a]	20–600	< 1	1–100	10*	1	1, 2, 3, 10
Rhodium–0.5 At.% Fe, encased	4–300	< 1	1–100	10*	1	3
Carbon resistance	10^{-2}–50	$\sim 10^{-2}$	< 10	10*	1	4, 5, 6
Germanium[b], encased	< 0, 1–100	10^{-1}	$\lesssim 0,1$	10*	0.1	7, 8, 13, 10
Thermistor	< 1–400	10^{-2}	< 10	10*	1	6, 9
● Semiconductor diodes, GaAs and Si	< 1–400	10^{-2}	< 1	10*	1	10, 14
● Capacitor thermometer, $SrTiO_3$	10^{-2}–60	10^{-1}	→ 0	10*	1	10
● Thermoelements						
Cu–constantan	50–800	10^{-4}	→ 0	**	$\delta T/T$: 0.5%	1, 2, 11
Cu–AuCo 2.1 At.%	20–300	10^{-4}	→ 0	**	1%	1, 2, 11
Chromel–AuFe 0.03–0.07 At.%	1–500	10^{-4}	→ 0	**	0.5%	1, 2, 11
Normal silver[c]–AuFe 0.03 At.%	1–20	10^{-4}	→ 0	**	0.5%	1, 2, 11
● Vapour pressure thermometer[d] ^3He	1, 0–3, 3	1	→ 0	10*	1	6, 12
^4He	1, 6–5, 0					
H_2	13, 9–23					

* Dependent on the method of calibration
** No absolute measurement
[a] With helium as exchange gas
[b] Doped with As, Sb, In or Ga
[c] Ag + 0.37 At.% Au
[d] The range of measurement corresponds to vapour pressures between 10 mbar and 2 bar for ^4He and H_2 or 1.1 bar for ^3He. For further filling gases, see [182, 183]

Sources of supply:
1. Heraeus
2. Degussa
3. Rosemount Engineering
4. Allen-Bradley
5. Speer Carbon
6. Leybold
7. Cryocal
8. Solitron
9. Keystone Carbon
10. Lake Shore Cryotronics
11. Sigm. Cohn, Mt. Vernon
12. Wallace Tiernan
13. Honeywell
14. Institute of Cryogenics, Southampton

- The most generally used sensors are based on the change with temp.:
  - of the resistance of various metals and carbon (*)
  - " " " semiconductors (Ge)
  - the emf generated between dissimilar metals (thermocouples)
  - the capacity of dielectrics ($Sr\ Ti\ O_3$)

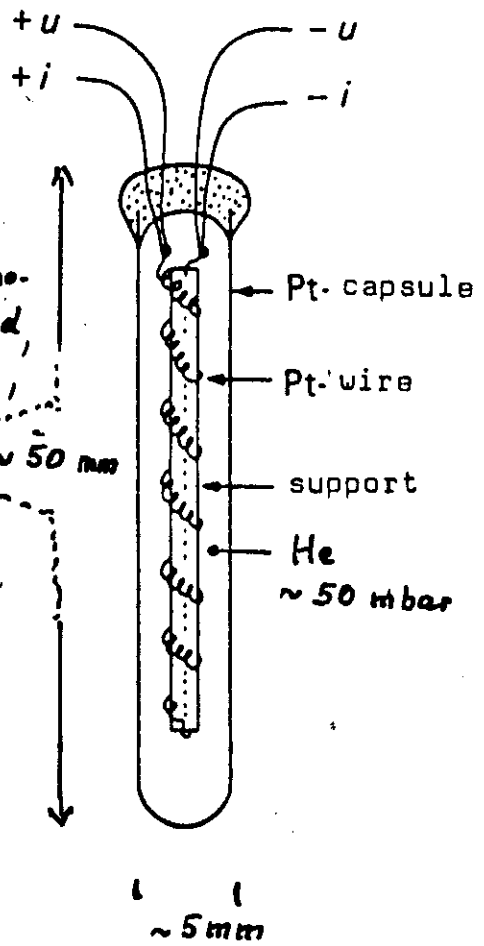
  Vapour pressure thermometry is not widely used in applications.
- Signal detectors can be: voltmeters, potentiometers or bridges, the latter two with AC or DC configurations.
- The method of conversion from signal into temperature can vary from the use of a simple curve or table to a dedicated microprocessor or main frame computer calculation.

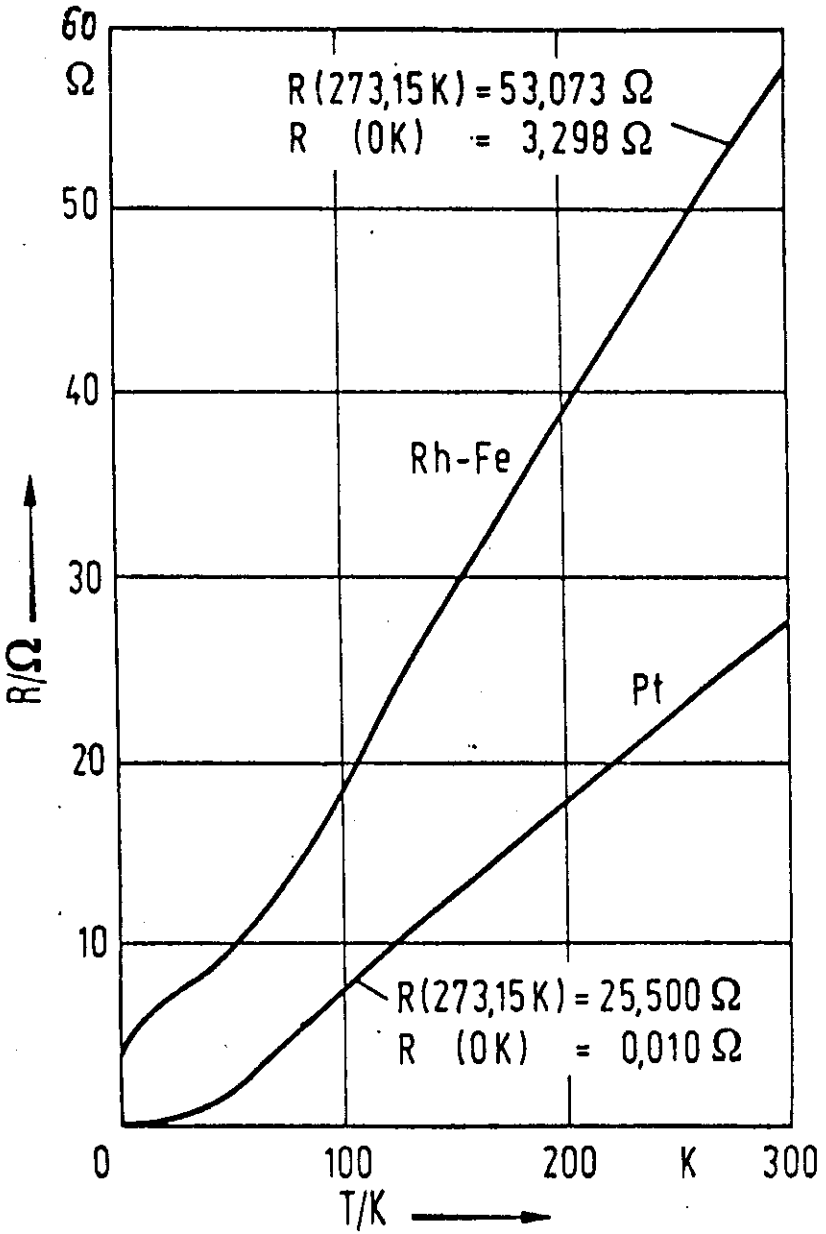
(*) The resistance of the lead wires can be eliminated by a three or four wire system. 

Pt-wire 0.1 mm ϕ
helix ~ 0,5 mm ϕ
support ~ 5 mm

Measurement range:
20 - 600 K

The Pt-resistance thermometer is the standard, i.e. the realization of the 1968 Int. temp. scale (IPTS 68) below 630.74 °C.

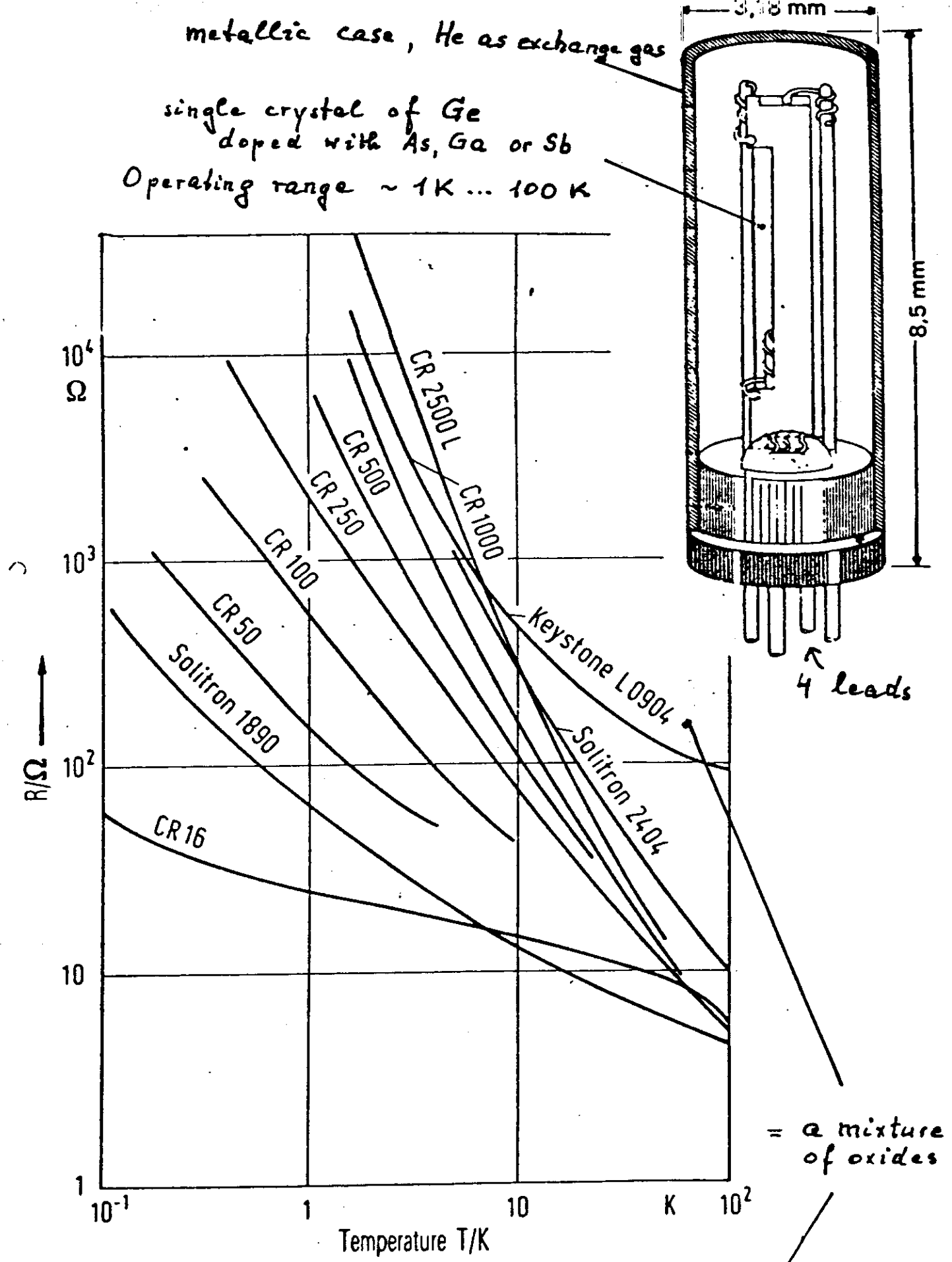




Measurement range
4 - 300 K

Rhodium +
0.5 At.% Fe

FIG. 9.45. *R,T* characteristic curves of a Pt and a Rh–Fe thermometer.[1]



R,T characteristic curves of some germanium thermometers, Solitron and Cryocal (CR) types, and of a Keystone thermistor.

They are composed of graphite in the form of a thin film.

Measurement range: ~ 0.1 – 100 K

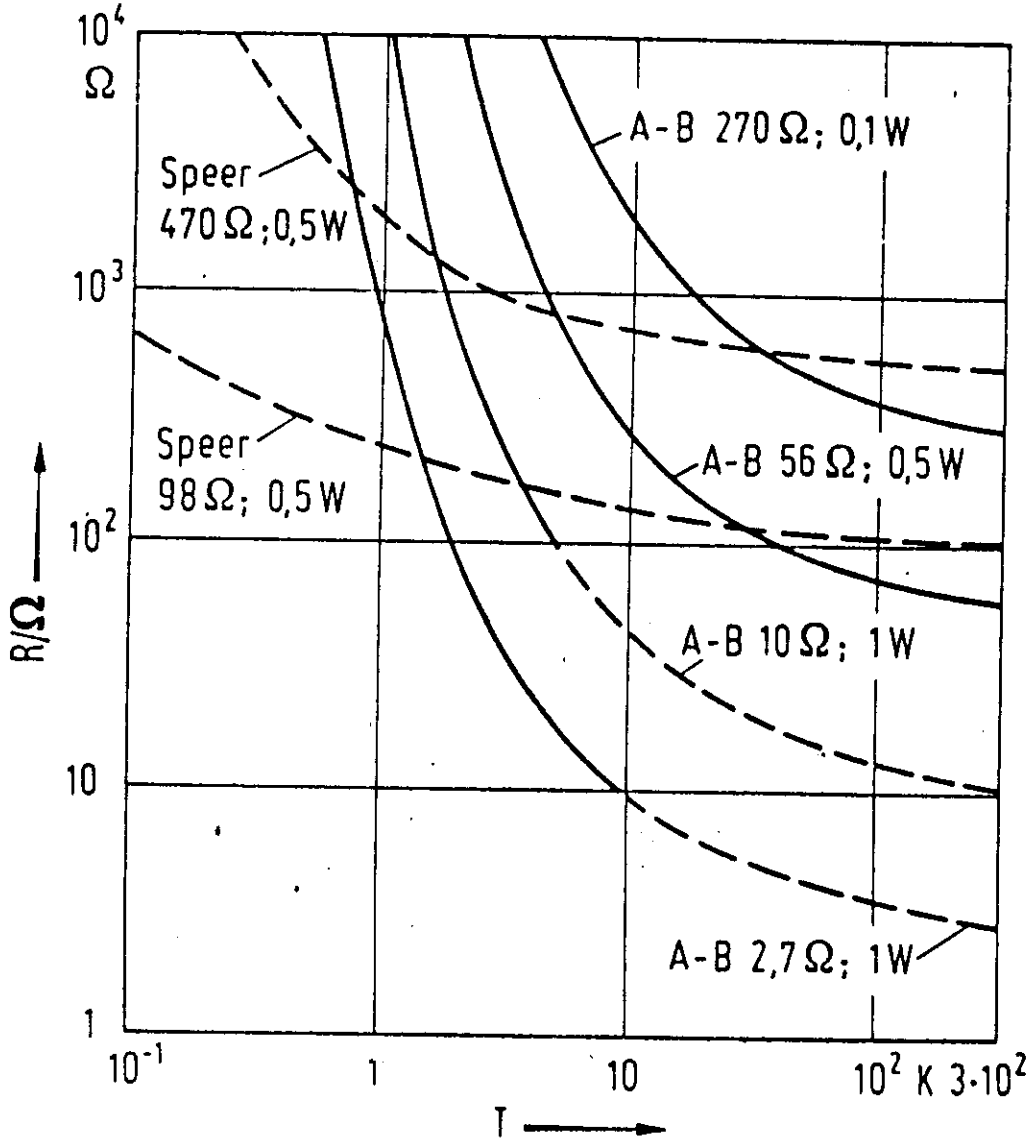
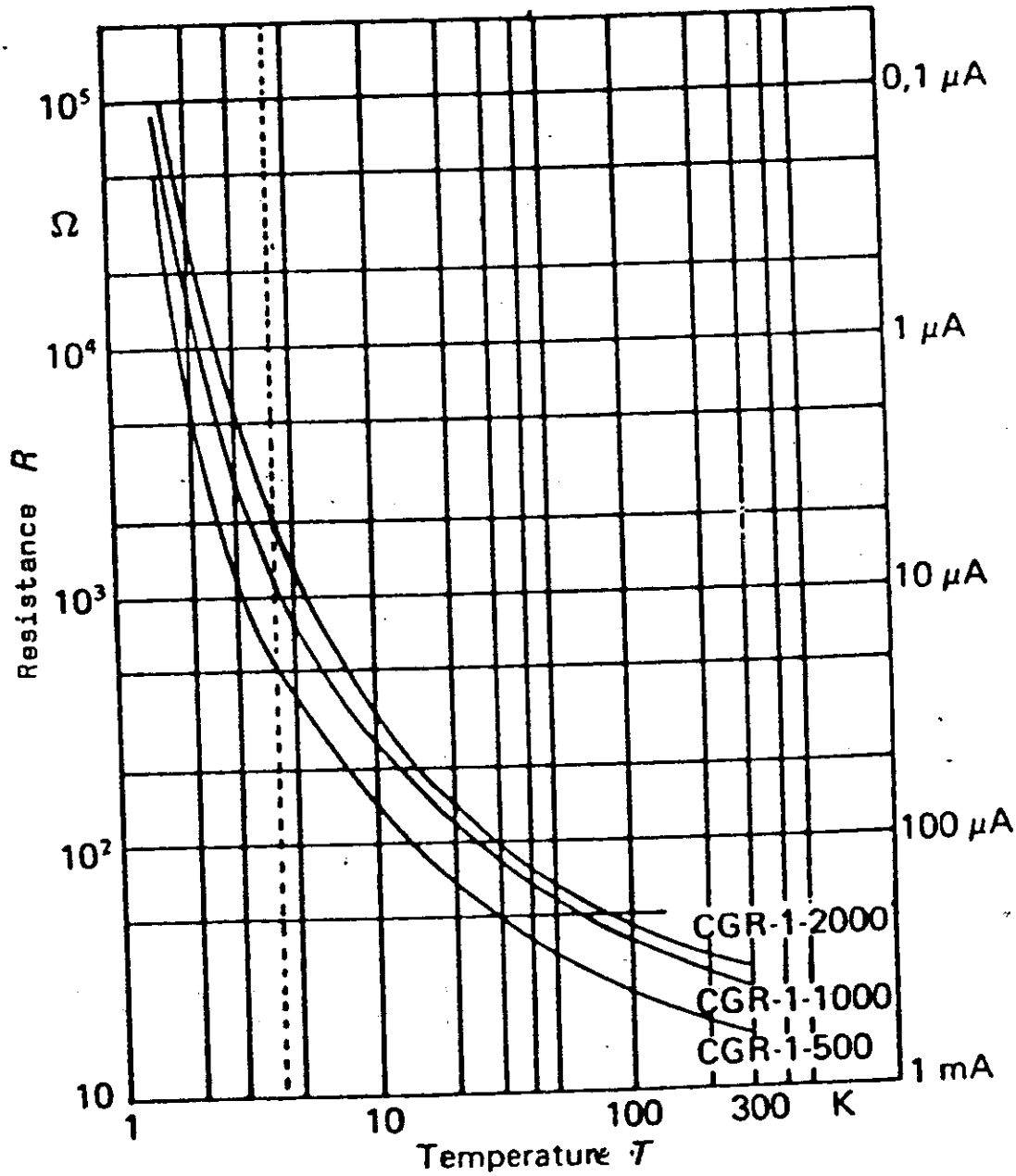


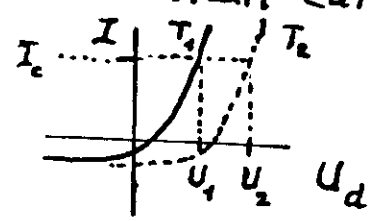
FIG. 9.46. R,T characteristic curves of some carbon resistance thermometers, Allen–Bradley (A–B) and Speer types.

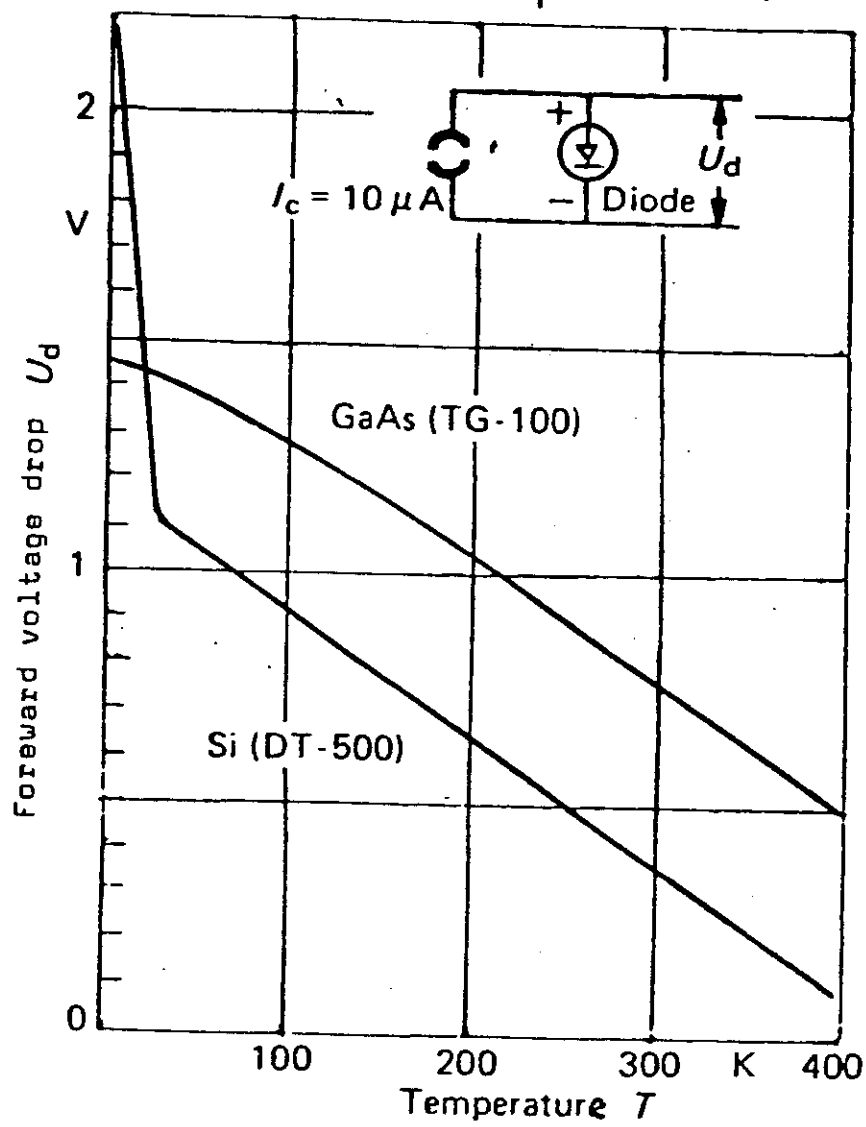
- They consist of carbon filaments deposited in the voids of a leached (porous) borosilicate glas.
- Sensitivity is a little higher and the reproducibility of the encapsulated models is better than those of A-B resistors.
- Operation range < 1 ... 300 K



R,T characteristic curves and recommended measuring current (right-hand scale) of carbon-in-glass temperature sensors.
Type: Lake Shore Cryotronics.

Diode temp. sensors The forward voltage drop across a p-n junction diode carrying a constant current increases as the temp. drops.

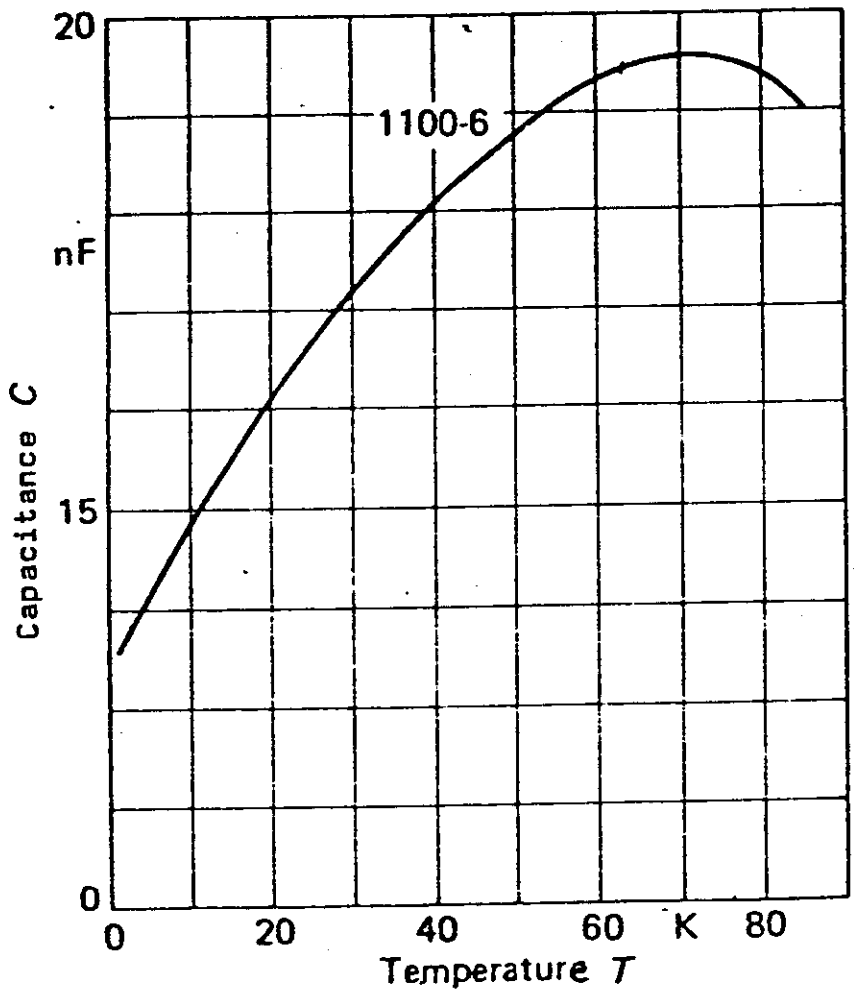


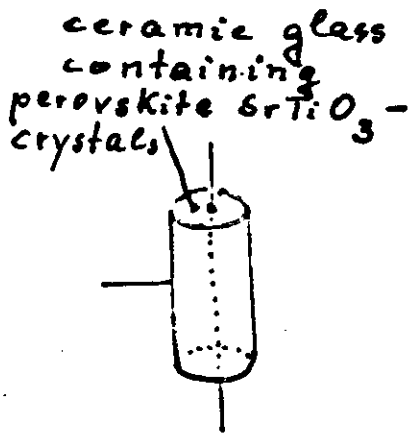


U_D measured by digital voltmeter

U,T characteristic curves of Si- and GaAs-diode thermometer sensors, Lake Shore Cryotronics.

(excepting vapour pressure therm.) are affected by magnetic fields as they depend on electron motion.
The dielectric constant is not so affected:
No dependence on magnetic field up to 14 Tesla (= 140 000 Gauss) within the measurement uncertainty of ± 1 mK.
Measuring range: 0.1 ... 70 K



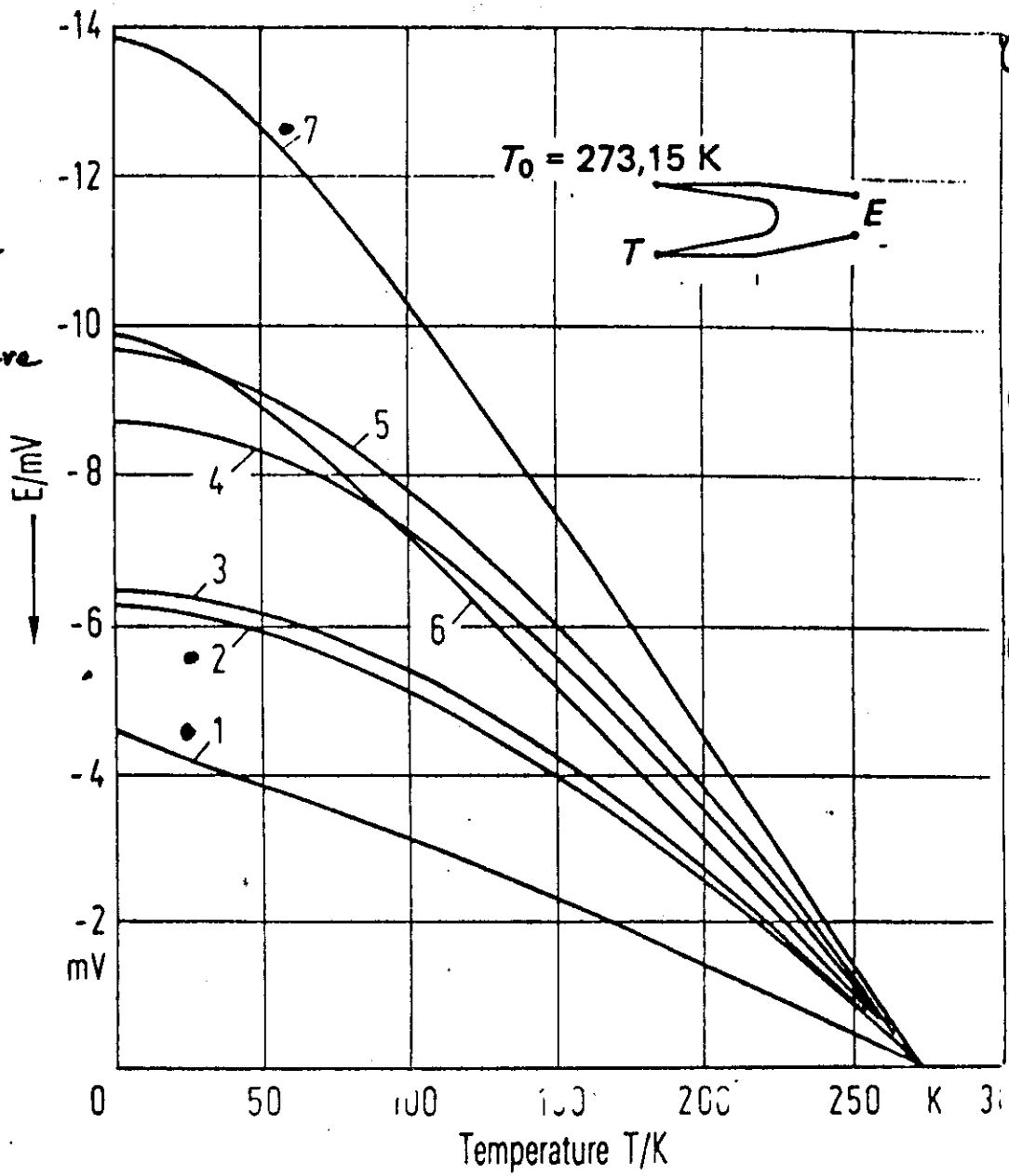


C,T characteristic curve of $SrTiO_3$-temperature sensor, Type Lake Shore Cryotronics.

The thermoelectric voltage can be measured with a digital voltmeter.

It is advantageous to have the reference junction of the thermocouple also at low temp., e.g. in LN_2 or LHe.

But there are also Peltier elements for the ice point 273.15K available.



(7) • NiCr - Au Co 2.1 % high thermoelectric but metallurgically unstable.

(2) • Cu - Constantan insensitive at T <

(1) • Chromel - Au Fe 0.03 metallurgically sta used successfully down to 1K

FIG. 9.48. Thermal e.m.f. of low temperature thermocouples.[185] (1) **Chromel-AuFe** 0.03. (2) Copper–constantan. (3) Chromel–Alumel. (4) Iron- Constantan Nickel chromium–constantan. (6) Copper–AuCo 2.1. (7) Nickel chromium-AuCo 2.1. $T_0 = 273.15$ K.

The cold heads of the two-stage refrigerators are provided as standard with a H_2-filled vapour pressure thermometer in the second stage.

This thermometer consists of a measuring sensor of volume V_M containing – at operating temp. – the condensed hydrogen. This volume is connected by the shielded capillary K to the manometer M, having a range from < 0.1 to 2 bar, corresponding a temp. range from 14 to 23 K.

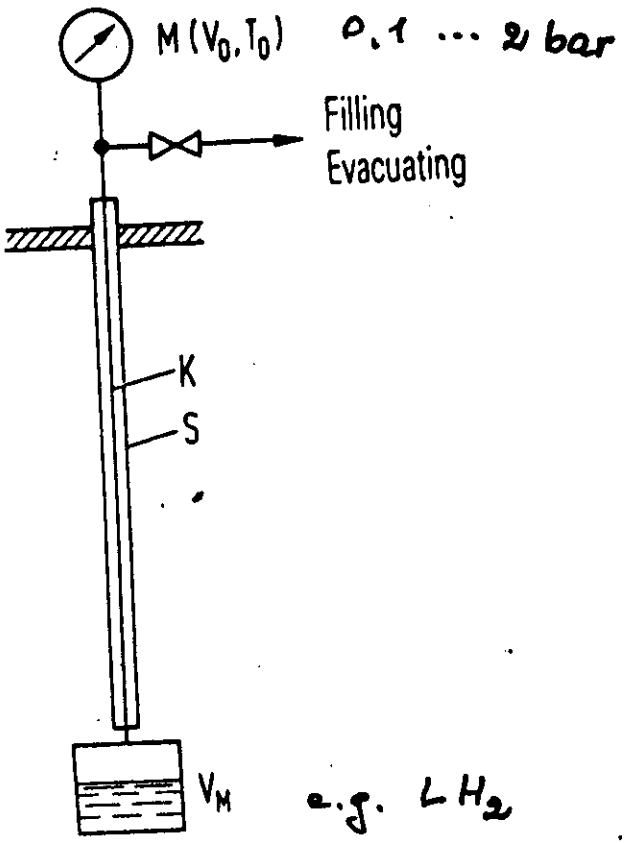


FIG. 9.49. A vapour pressure thermometer (schematic). M, Manometer of volume V_0 at temperature T_0. K, Capillary. S, Shielding. V_M, Volume of the sensor.

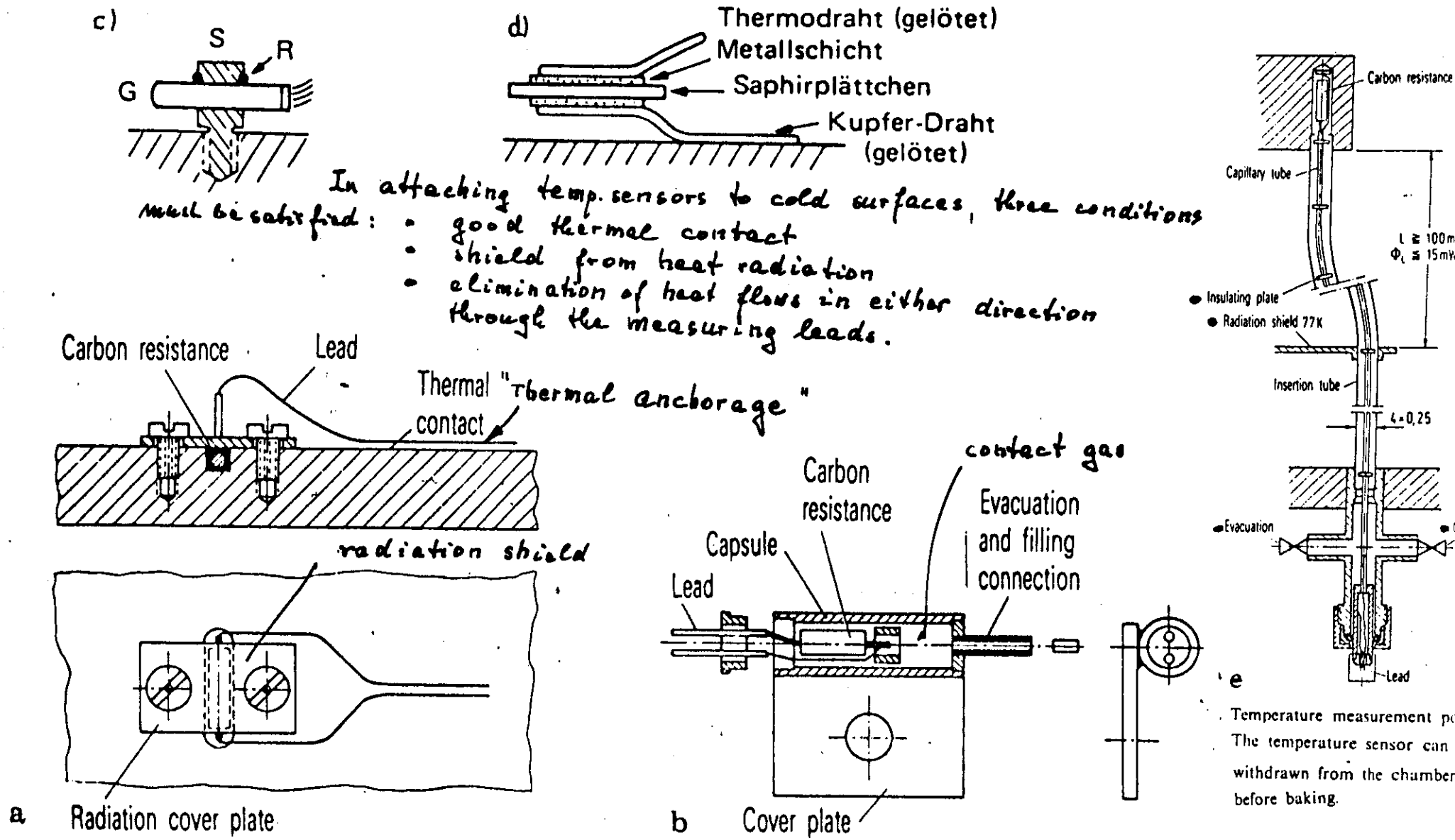


FIG. 9.50. Temperature measurement points. The temperature sensor is (a) fixed with adhesive, or (b) encased in a capsule with exchange gas. After Walter[206] (source: Fritz-Haber-Institute, Berlin).

TABLE A3. Properties of refrigerants used in cryogenic technology [A1–A11]

Refrigerant			^{3}He	^{4}He	c-H_2	n-H_2
Molar mass	M	kg kmol^{-1}	3.0160	4.0026	2.0159	2.0159
Boiling point at 1.01325 bar	T_b	K	3.190	4.222	20.27	20.397
Critical point						
Temperature	T_c	K	3.33	5.22	32.98	33.24
Pressure	p_c	bar	1.16	2.30	12.93	12.98
$RT_c\rho_c/Mp_c$	z_c	—	3.308	3.262	3.304	3.179
T_b/T_c		—	0.96	0.81	0.61	0.61
Triple point				λ-point		
Temperature	T_t	K		2.173	13.80	13.956
Pressure	p_t	mbar		50.52	70.4	72.0
Density						
Gas, 273 K, 1 bar	ρ_N	kg m^{-3}	0.13272	0.17614	0.08868	0.08868
Critical point	ρ_c	kg m^{-3}	41.8	69.5	31.4	30.1
Vapour at T_b	ρ''	kg m^{-3}	27.3	17.2	13.4	13.3
Liquid at T_b	ρ'	kg m^{-3}	58.9	124.8	70.8	70.8
Solid at T_t at zero point	ρ_t	kg m^{-3}			87	
$T=0$ $p=0$	ρ_0	kg m^{-3}		146	88.4	
Vol. gas 273 K 1 bar from 1 m^3 liquid	ρ'/ρ_N	m^{3}m^{-3}	444	709	798	798
Latent heat						
Evaporation at T_b	l_v	kJ kg^{-1}	15.87	20.91	446.5	448.3
Melting at T_t	l_t	kJ kg^{-1}	(at 1.65K)	5.22 (103 bar)	58.04	58.04
Evaporation entropy	Ml_v/T_b	kJ kmol^{-1}K^{-1}	14.9	19.9	44.4	44.4
Specific heat capacity						
Gas, 273 K, 1 bar	c_p	kJ kg^{-1}K^{-1}	6.89	5.233		14.21
Vapour at T_b	c_p	kJ kg^{-1}K^{-1}	6.89	5.23	10.31	10.31
Liquid at T_b	c_p	kJ kg^{-1}K^{-1}		4.410	9.30	9.28
Solid at T_t	c_p	kJ kg^{-1}K^{-1}			2.90	3.03
Debye temperature	Θ_D	K		29 (T < 0.7 K)		
c_p/c_v 1 bar, 300 K	κ	—	1.66	1.660	1.405	1.405
Vapour enthalpy	$h_{300}-h_{T_b}$	kJ kg^{-1}		1541.8		3509.5
Minimum liquefaction work	e_L	kJ kg^{-1}		6819		12019

c-D_2	n-D_2	Ne	N_2	CO	Ar'	O_2	CH_4	Kr
4.0282	4.0282	20.183	28.013	28.000	39.948	31.999	16.043	83.80
23.64	23.67	27.102	77.348	81.67	87.26	90.188	111.67	119.74
38.26	38.35	44.39	125.98	132.91	150.68	154.78	190.7	209.7
16.49	16.65	27.21	33.93	34.99	48.63	50.80	46.40	54.83
3.200	3.202	3.250	3.426	3.397	3.424	3.008	2.942	3.448
0.62	0.62	0.61	0.62	0.62	0.58	0.59	0.52	0.57
18.69	18.73	24.559	63.148	68.09	83.82	54.361	90.67	115.94
171.3	171.5	433.0	126.1	153.7	687.5	1.52	116.7	731.9
0.1773	0.1773	0.8881	1.2342	1.2337	1.7606	1.4101	0.7089	3.696
66.9	67.4	483.5	311	301	530.8	380	138.1	908.5
		9.46	4.59	4.445	5.72	4.44	1.825	8.33
166	166	1204	804.2	793	1399	1140	424	2413
			946		1622			
		1442	1137		1707	1568		3186
937	937	1356	652	643	795	808	598	653
303.6	304.4	87.20	199.1	215.9	163.2	213.1	510.3	107.7
48.91	48.91	16.60	25.73	29.86	29.44	13.88	58.72	19.52
51.6	51.6	64.6	72.0	74.0	74.6	74.5	73.5	75.2
	7.25	1.030	1.042	1.042	0.521	0.918	2.235	0.248
5.18	5.16	1.030	1.039	1.036	0.521	0.909	1.870	0.248
6.50	6.12	1.84	2.03	2.21	1.05	1.70	3.42	0.538
2.96	2.88	1.302	1.691		0.833	1.444		0.428
89	89	64	68		80	90.9		63
(T < 13 K)	(T < 12 K)	(T < 12 K)	(T < 20 K)		(T < 10 K)	(T < 15 K)		(T < 10 K)
1.40	1.40	1.668	1.401	1.401	1.670	1.396	1.30	1.689
		283.46	233.8	228.8	111.3	193.2	403.5	
		1376	766	756	480	638	1109	

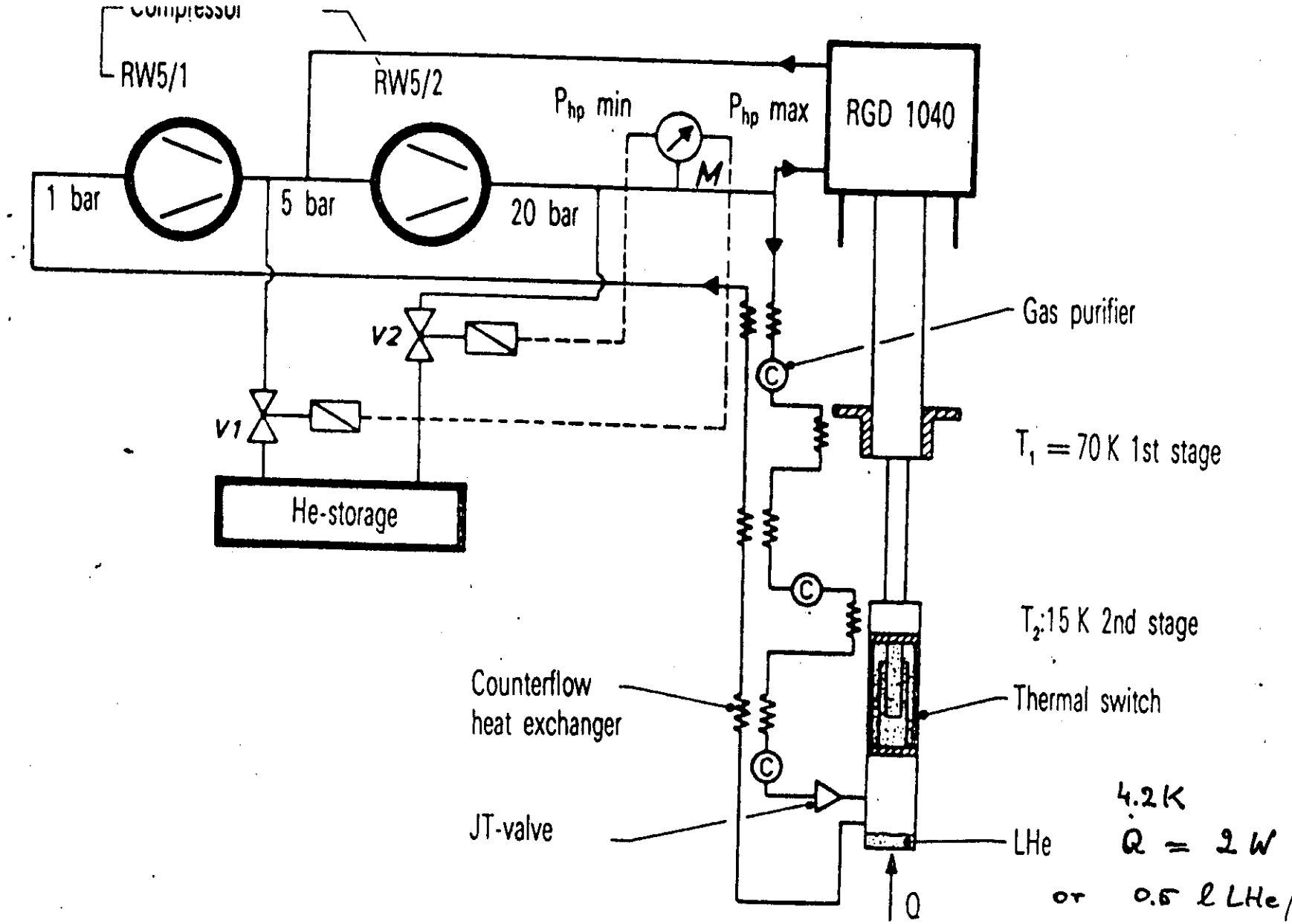


FIG. 10.57. A three-stage refrigerator, type Leybold RGT, equipped with standard two stage coldhead RGD 1040 and Joule Thomson stage JT.[400] (Courtesy of Leybold AG.)

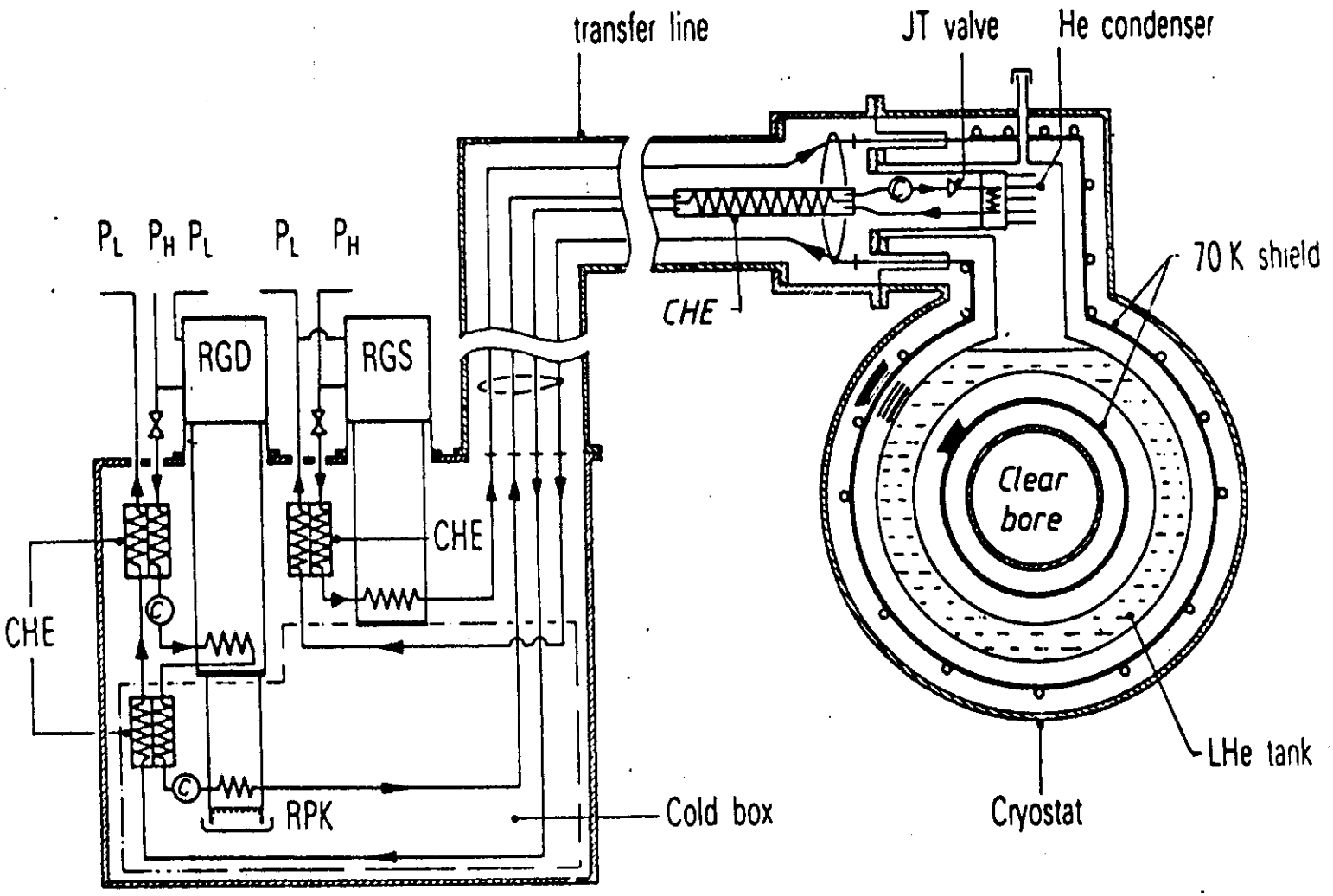


FIG. 10.61. Cold box refrigeration system with helium recondensation, type Leybold.[404] (Courtesy of Leybold AG, Cologne.) RGD, standard two-stage coldhead. RGS, standard single-stage coldhead. CHE, counterflow heat exchanger. C, gas purifier (charcoal adsorber). RPK, cryopump array.