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K-Series Centrifuges

IV. Measurement and Control of Temperature

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The K-series centrifuges were initially designed for the isolation of influenza virus from chorioallantoic fluid (1, 2). Temperature control is not critical for the preparation of influenza vaccine; therefore, provisions for it were minimal. Aqueous coolant in a containment system was circulated around the seals, cooled oil was passed through the bearings, and cold brine was passed through a refrigeration jacket lining the rotor chamber (2).

The use of the K-series centrifuges has been expanded beyond vaccine production into the field of serum and subcellular particle fractionation by the design and fabrication of rotors K-III through K-XI (to be described in later publications). For many of these separations it is necessary to control the temperature of the rotor and the continuous-flow sample stream near 3°C in a K-centrifuge with a C-type casing (1).

SYSTEM DESCRIPTION

The thermodynamic system can be divided into two parts (Fig. 1): (*a*) the rotor secured inside an evacuated armor casing by two hollow axial shafts and (*b*) the sample fluid lines which consist of a hollow passage through the lower seal housing, the lower seal, the lower shaft, the upper shaft, the upper seal, and the upper seal housing. The lower shaft is supported by one journal bearing, whereas the upper shaft passes through three journal bearings and the turbine wheel which is mounted on two ball bearings.

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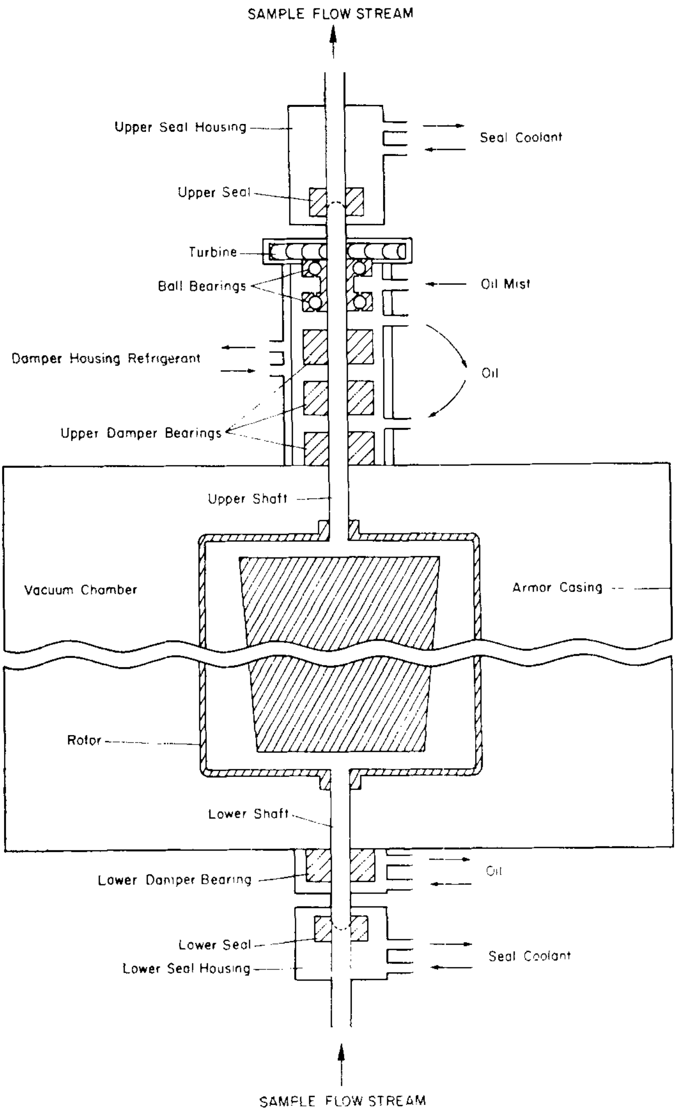

FIG. 1. Sample flow stream of K-series centrifuge with associated components.

Sources of heat in the system. The division of the thermodynamic system into two parts reflects the two basic operating modes of the centrifuge, namely, batch and continuous-sample flow. When the centrifuge is operated with a batch-type rotor that is loaded and unloaded at rest (statically), only the rotor temperature is of concern during rotation. In this mode, heat enters the system by conduction along the two shafts, conduction across the armor casing with radiation and gas conduction to the rotor, and from collision of gas molecules with the rotor surface. The second basic use of the centrifuge is continuous-flow operation. Here the sample stream flows into the spinning rotor where the particles of interest (or contaminating particles) are removed from the flow stream which then continues on out of the centrifuge. In this operation it is necessary to control the temperature of the continuous-flow stream as well as the rotor temperature. Therefore, to the heat sources previously considered must be added friction in the four journal bearings, in the ball bearing supports of the turbine wheel, and between the seal faces. This heat is

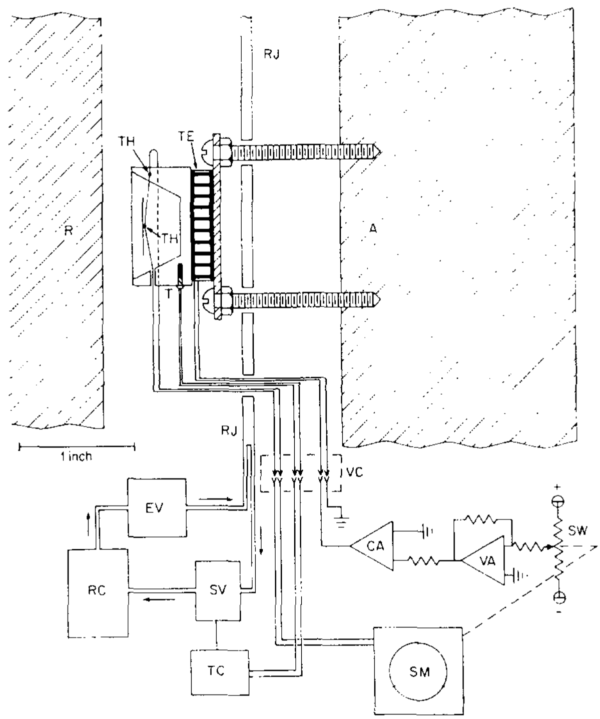

FIG. 2. Temperature detection and control instrumentation for K-C centrifuge:

RC, refrigeration compressor
EV, expansion valve
RJ, refrigeration jacket
SV, solenoid valve
TC, temperature controller
R, rotor
TH, thermocouples
VC, vacuum coupling
SM, indicating potentiometer
SW, retransmitting slide wire
CA, current amplifier
TE, thermoelectric module
A, armor casing
T, thermistor
VA, voltage amplifier

conducted directly across the shaft wall to the sample stream, as is heat from the environment.

The problem is thus resolved into temperature determination of the rotor, dissipation of heat from its surface, and removal of heat from the seals and bearings with appropriate temperature measurement.

Rotor surface heat removal and temperature detection. The refrigeration jacket lining the rotor chamber was incorporated into a gas-expansion refrigeration system (Fig. 2). The system is controlled through a solenoid valve in the low pressure line of a 1 hp compressor in order to minimize overshoot of the rotor temperature. Freon 12 was the original refrigerant; however, a 3:1 mixture of Freon 12/Freon 22 was subsequently used to achieve lower jacket temperatures (—45°C).

The rotor temperature sensing device must accurately determine the rotor surface temperature (0 to +37°) while being in close proximity to the refrigeration jacket (0 to —45°). To prevent the refrigeration jacket temperature from biasing the rotor temperature measurement, a radiometer assembly similar to that used in the Beckman model L-4 ultracentrifuge[3] was employed. Two thermocouples were formed back-to-back by welding a short section of 36-gage constantan wire between two pieces of 36-gage copper wire. One thermocouple was cemented to the back of a 0.5 in. diameter disc of aluminum foil, the face of which had been blackened; the other was inserted into the body of a bright aluminum reflector in which the foil disc is mounted. Any difference in temperature between the disc and its reflector (and, therefore, between the two thermocouples) produces a net voltage. This voltage actuates a potentiometer with a range of —0.5 to +0.5 mV (ΔT = —12.8° to +12.8°C). A retransmitting slide wire in the potentiometer was connected between +15 and —15 V and its wiper tied to the sequence: operational amplifier, current amplifier, and a thermoelectric module[4] physically attached to the back of the reflector. Thus, a difference in temperature between the disc and its holder-reflector initiates a chain of events that results in the temperature of the reflector attaining that of the disc. Starting from equilibrium, a ΔT of 0.1°C is sufficient to activate the thermoelectric module. This assembly permits two options not otherwise open: (*a*) the temperature of the rotor can be accurately measured with a thermistor, which then controls[5] the refrigeration system, and (*b*) the measuring de-

[3] Beckman Instruments, Inc., Palo Alto, California.

[4] Model 3952-1, Cambridge Thermionic Corp., Cambridge, Mass. Mention of commercial products in this paper is meant to be informative rather than restrictive. Other products of similar characteristics may work equally well.

[5] Model 72 temperature controller, Yellow Springs Instruments, Yellow Springs, Ohio.

vice can be mounted close to the refrigeration jacket without affecting the accuracy of rotor temperature measurements. The thermistor was inserted in a hole in the reflector packed with heat sink compound,[6] and the hole was sealed with a metal epoxy cement in order to prevent electrical leakage due to oil. The entire assembly was mounted on an aluminum plate that was bolted to the armor ring through holes drilled in the refrigeration jacket, the armor ring being the heat sink for the thermoelectric module.

The accuracy of the radiometer assembly was tested against a thermistor attached to the surface of the stationary rotor with heat sink compound. Under a variety of cooling conditions there was no significant difference between the two readings as long as pressures lower than 50 μ Hg were maintained.

The efficiency of heat dissipation from different rotor surfaces was tested (Table 1). The clear anodized aluminum surface (K-II) was quite

TABLE 1
Heat Removal from Rotor Surface[a]

	Rotor				
	K-II	K-IIB	K-III	K-IIIB	
Refrigeration jacket (°C)	−25°	−25°	−25°	−25°	−45°
Temp. held when set to control at 3°C	3°	3°	6°[b]	3°	3°
Time to drop from 3° to 2°C	18 min	>90 min	—	62 min	17 min

[a] 30,000 rpm, 25 μ Hg, no fluid flow through rotor, oil and seal coolant at room temperature.
[b] Oil and seal coolant chilled.

satisfactory and the application of velvet black paint[7] (K-IIB) only acted as an insulator. The untreated titanium alloy surface (K-III) was clearly unsatisfactory. The application of a 0.003–0.005 in. thick bonded black epoxy coating[8] (K-IIIB) increased heat dissipation to the point that the rotor would hold 3°C, but control was marginal, as shown by the time required to reduce the temperature to 2°. At this point the refrigeration jacket temperature was lowered with improved control.

[6] No. 340 silicone heat sink compound, Dow Corning Corp., Electronics Products Division, Hemlock, Michigan.
[7] Nextel Velvet Coating 101-C10 Black, Reflective Products Division, Minnesota Mining and Manufacturing Co., St. Paul, Minnesota.
[8] HINAC BK-2, Peninsula Products Finishing Co., Redwood City, California.

Heat removal from bearings and seals. The initial attempt to cool the sample stream was to refrigerate the seal coolant and the lubricating oil. Thermocouples were installed to record the inlet and outlet temperatures of the sample stream, the lubricating oil to both bearing assemblies, and the coolant to each seal. This cooling procedure succeeded in decreasing the temperature differential of the sample stream ($T_{\text{effluent}} - T_{\text{influent}}$) at all flow rates (Fig. 3). It was further observed that the temperature dif-

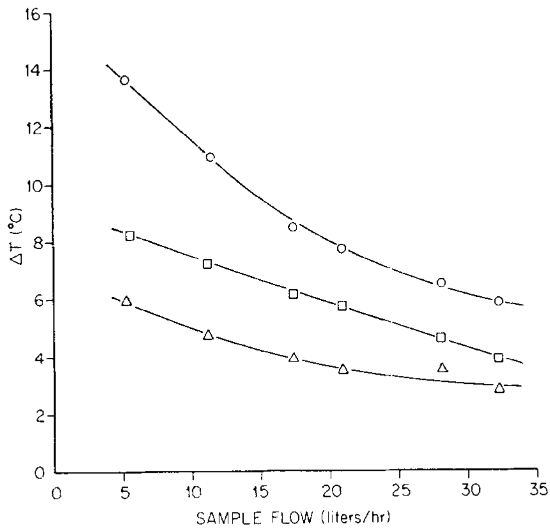

FIG. 3. Sample stream differential temperature as function of sample stream flow rate. Sample: water, 3°C input temperature. Rotor: K-IIIB, 30,000 rpm, temperature set to control at 3°. Ancillary cooling conditions: (○) seal coolant, 27°; oil, 27°; flow, bottom-to-top. (□) seal coolant, 8°; oil, 2°; flow, bottom-to-top. (△) seal coolant, 8°; oil, 2°; flow, top-to-bottom.

ferential was less if the sample flowed in the top and out the bottom than if the stream flowed upward. We interpret this to be due to the axial distribution of the heat load. The lower shaft (low heat load) runs in a single journal bearing, whereas the upper shaft (high heat load) runs in three journal bearings and is attached to the turbine with its two ball bearings. Thus, fluid entering the rotor from the top will carry the heat absorbed from the upper bearings into the rotor, from which it can be dissipated to the refrigeration jacket, while, in upward flow, the heat from the upper bearings will leave the system directly in the effluent stream with only the seal coolant available to lower the stream temperature. This interpretation is supported by these facts: when the refrigeration jacket was run at −25°C, the rotor temperature increased during top-to-bottom flow in the black titanium rotor. However, when the jacket was run at −45°, the rotor temperature was controlled within ±1°C. Second, the temperature rise of the sample stream in bottom-to-top flows is dependent on the seal coolant temperature (Fig. 4). The dependence is less marked in top-to-bottom flow because the lower seal housing provides a smaller heat exchanger and there is a lesser temperature differential (due to the single bearing).

The lubricating oil[9] originally used for the damper bearings was specified for operation at 25–50°C. Cooling to 0° would increase its viscosity such that heating would result in the bearings. Several oils that had desirable temperature-viscosity characteristics were tested for inducing swelling of the silicone-rubber damper pads. An important consideration

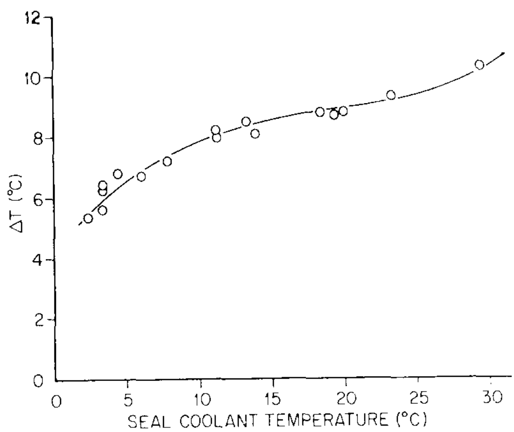

FIG. 4. Sample stream differential temperature as function of seal coolant temperature. Sample: water, 3°C input temperature, bottom-to-top flow at 18 liter/hr. Rotor: K-IIIB, 30,000 rpm, temperature set to control at 3°. Ancillary cooling conditions: oil, 20°.

is to find a lubricant with a satisfactory viscosity at low temperatures that retains sufficient lubricity at 50–75° should a refrigeration unit fail during high-speed operation. Mobil DTE-797[10] has been used while we are further testing several hydraulic fluids rich in polyisobutylene. Figure 5 shows that, at oil inlet temperatures of 0–17° (Mobil DTE-797), the temperature differential of the sample stream is practically unchanged.

The cooling capacity of the oil flow was improved by modifying the

[9] Esso Aviation 65, Humble Oil and Refining Co., Houston, Texas.
[10] Mobil Oil Co., New York, New York.

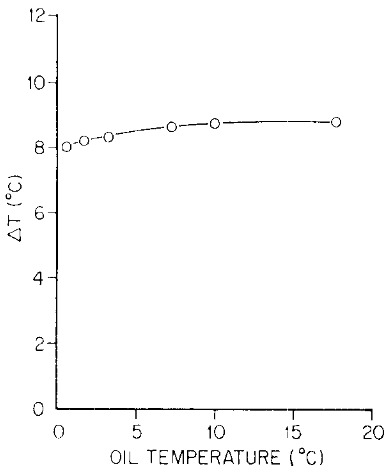

FIG. 5. Sample stream differential temperature as function of oil temperature. Sample and rotor: as in Fig. 4. Ancillary cooling conditions: seal coolant, 20°C.

damper housing to provide positive oil flow through the two upper bearings instead of only through the top bearing, as originally provided. This resulted in a 20% decrease in the sample stream temperature differential. However, we have since replaced the entire damper housing with a fluid-cooled model,[11] which when used with antifreeze at −5° makes refrigeration of the oil reservoir unnecessary.

CONCLUSION

The use of the K-centrifuge has been extended to temperature-sensitive material through the use of a radiometer for temperature detection and a gas-expansion refrigeration jacket for rotor temperature control. The sample stream differential temperature has been lowered to a usable range through refrigeration of the seal coolant and the damper housing. If a temperature-sensitive particle is to be collected in the rotor during continuous-flow operation, it would be preferable to direct flow from bottom-to-top; thus, the particle does not pass through the high heat load area. Conversely, if a particle is to remain in the sample stream, i.e., preparation of a high-speed supernatant, two options are open: bottom-to-top flow with rapid cooling of effluent, either with an efficient heat exchanger in the outflow line, or of the bulk solution; or top-to-bottom flow and allowing the rotor itself to act as a heat exchanger.

[11] No. SK-7-29-58 water cooled damper housing, Barbour-Stockwell Co., Cambridge, Massachusetts.

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