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Designing Aspects of Cryogenic Attachment For Uv/Visible Spectrophotomer

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ABSTRACT

The present paper deals with designing a cryocooling attachment for Ultra visible / visible spectrophotometer for spectroscopic analysis of materials at cryogenic temperature. The cryogenic environment can be provided to the sample holder of UV/ visible spectrophotometer with the help of cold finger made of copper rod, dipped in the liquid nitrogen vessel. For this, a liquid nitrogen vessel has been designed, kept in a vacuum chamber to prevent any ambient effect.. The performance can be evaluated by comparing the images produced by UV/ visible spectrophotometer for ambient and cryogenic environment as well as the temperature reading of the different samples, under observation.

Keywords : UV/VISIBLE SPECTROPHOTOMETER, LIQUID NITROGEN TEMPERATURE, COLD FINGER, CRYOGENIC VESSEL

INTRODUCTION:
Temperature is one of the important parameters that one may vary during optical spectroscopic measurements. Most of emission and absorption lines are broadened due to thermal motion: cooling allows to reduce line widths and to discriminate between lines, which are spectrally close to each other. At cryogenic temperatures, absorption features are usually narrower and more sharply resolved, sample degradation is slowed, and it is often possible to trap unstable intermediates. Liquid helium experiments are complex and expensive, and liquid nitrogen temperature (around 77K) suffices for many applications.

An attachment for low temperature scanning optical microscope has been designed (Sang-Youp Yim, 2010). In order to scan the sample, they connect the sample holder to the cryostat cold finger by using a flexible gold braid LTOM system can operate in any type of configuration, such as illumination, collection or reflection mode.

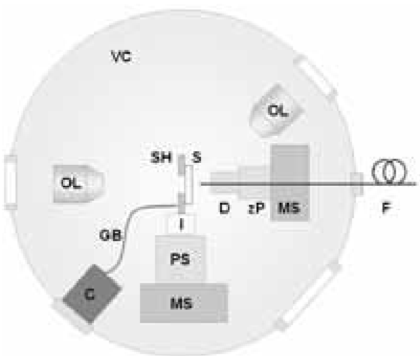

Figure 1 Schematic diagram of the LTOM scan head in a vacuum chamber (top view)

Shown are the (C) cold finger, (D) dither piezo, (F) fiber probe, (GB) gold braid, (I) insulator, (MS) motorized stage, (OL) objective lens, (PS) piezo scan stage (x-y), (S) sample, (SH) sample holder, (VC) vacuum chamber, and (zP) z piezo, three optical view ports (OP).

The Helitran (ARS Cryo, 2010) has designed an attachment for micro spectroscopy and has some unique features not found in other similar cryostats. It is the only cryostat made of welded stainless steel for cleaner sample environment; this means no water condensation on the sample surface. No water peaks. The second unique feature is the infinite adjustment of sample to window distance (screw mounting arrangement), so no matter how thick the sample is, it can be kept as close to the inner surface of the window.

2. DESIGN METHODOLOGY: The commercially available U-V visible spectrophotometer work at ambient conditions. This instrument can do wonders if it works at cryogenic temperature. So the aim of this work is

- to design liquid nitrogen vessel
- Cryogenic temperature of 77K to 100K is provided by cold finger of copper rod dipped in LN2 vessel and extended to the sample holder located in U-V visible spectrophotometer.
- to develop a system which is easily accessible, free from magnetic interference and vibration.
- the frosting on the and heat losses from the sample and the weight of the system will be as minimum as possible
- system will retain liquid nitrogen until the spectrophotometer carry out its function.

2.1 LIQUID NITROGEN VESSEL DESIGN:
The best material to fabricate LN2 vessel is SS304 due to its

- Strength
- Corrosion resistance
- Fracture hardness
- Good machinability
- High ductility
- Essentially non-magnetic, becomes slightly magnetic when cold worked

2.2 INNER AND OUTER VESSEL DESIGN (C.A.Bailey & B.A.Hands, 1986)
A cylindrical vessel is most economical and easier from the fabrication point of view and ideal for transportation. The wall thickness of the cylindrical vessel should withstand internal pressure. Pressure of the vessel can be calculated using the Lame's formula as follows:

$$P = S(D^2 - d^2) / (D^2 + d^2)$$

Where: D= nominal outside diameter

d= nominal inside diameter

p= design internal pressure

Sa = allowable stress of material at 4:1 design factor, 129.2 MPa

Schedule 5 pipes are selected because they are the thinnest pipes available in SS 304 class. According to the ASME Code, Section VIII, the minimum thickness of the inner shell for a cylindrical vessel should be determined from equation;

$$t = P_i \times d / [2 S_a \times e_w - 1.2 P_i]$$

where, t = Thickness of the vessel,

D_i = the inside diameter of the vessel,

p= design internal pressure,\

s_a = Allowable stress,

e_w = weld efficiency

Assuming pressure inside the vessel = 3 atmospheric pressure and weld efficiency, e_w = 1 is safe criteria from design point of view.

2.3 LIQUID NITROGEN FILLING VOLUME:
Volume for inner cylinder
$=[\pi \times Di^2 \times \text{height}] / 4$

This is necessary to calculate as it will give idea about the capacity of the vessel and consequently help to find out the time required for refilling liquid nitrogen, if need arises.

2.4 LANGE FOR ASSEMBLY:
Flange of suitable diameter is selected keeping in mind for accommodation of vent and fill line.

2.5 SUSPENSION SYSTEM:
No suspension system required in this vessel because vessel is very small.

2.6 PIPING:
Piping necessary to fill liquid to the container and to vent vapor from the vessel, introduces a source of heat in-leak to the container. With a properly designed piping system, the heat transfer can be reduced by using thin walled pipes. The minimum wall thickness for piping subjected to internal pressure is determined according to the ASA Code for Pressure Piping by the following expression:

$$T = p\, D\, o / (2sa + 0.8p)$$

where p = design pressure = assume 3 atm. Pr.

Table 1 Different Component Dimensions

Component	Dimensions, mm
Inner Vessel	Dia. 42.2, L = 120
Outer vessel	Dia. 48.3, L = 127
Fill and vent port	Dia. 8 , L = 80
Flange	Dia. 52, T = 3

2.7 INSULATION (R.F.Baron,1985) : Vacuum insulation alone is used extensively for small laboratory-size dewars. The use of Vacuum insulation essentially eliminates two modes heat transfer; solid conduction and gaseous convection

Advantages of vacuum insulation:-
- Small cool-down loss.
- Low heat flux for small thickness between inner and outer vessel
- Complicated shapes may be easily insulated
- Low cost compared to MLI and opacified powder insulation
- No stringent requirement of vacuum level

2.8 HEAT TRANSFER CALCULATIONS (C.A.Bailey & B.A.Hands, 1986)
Heat Transfer through Insulation
The radiant heat –transfer rate between two surfaces is given by the modified Stefan-Boltzmann equation:

$$Q = F_e F_{1-2} \sigma A_1 (T_1 - T_2)^4$$

where, Fe = Emissivity factor. It is given by, $1/F_e = 1/e_1 + A_1/A_2 [1/e_2 - 1]$

e_1, e_2= the emissivity of the surface at 77 K and 300 K respectively.

σ = Stefan-Boltzmann constant = 5.679×10^{-8} W/m^2-K^4

A_1 , A_2 = area of inner and outer vessel surface rerspectively

F_{1-2} = configuration factor =1

For two concentric vessels, F1-2 =1.

Heat Transfer through pipe line:
$Qp = (Ka - Ki) A_p / L$

Where A_p is cross section area of piping

Ka = Thermal conductivity of SS-304 at ambient temperature = 14.90 W/m K

Ki = Thermal conductivity of SS304 at NBP of LN_2 = 8.26 W/m K

To reduce heat transfer through top flange, it can be covered by Teflon.

$Q_F = Q_{cond} + Q_{conv}$

$= \Delta T / R_t + h_c A_f \Delta T$

where $R_t = 1 / A [\delta / K]$

δ = thickness of insulation

K = thermal conductivity of Teflon

= 0.23 W/mK

hc = convective heat transfer coefficient of liquid nitrogen = 32 W / m^2 K

Table 2 Calculated Heat Transfer through Different Components

Component	Heat Transfer, watts
Insulation	3.664
Piping	1.305
Flange	69.43
Vacuum Chamber	21.94

Total Heat transfer: In calculating total heat transfer, heat load of vacuum chamber also have to be accounted.

So, total $Q = Qi + {}^{Q}F + Q_{v.c.} + Qp$

Boil- off rate of Liquid Nitrogen:
Now , $Q = m_{LN2} \times h_{fg}$

Where h_{fg} = Latent heat of vaporization of LN_2 = 200 KW

So, $Q = m_{LN2} \times 200 \times 10^3$ W

Therefore, we get the mass of liquid nitrogen required (kg /s)

As 1 kg = 1.098 Liter, we can have the value of mass of liquid nitrogen in ml / sec.

As the filling volume for liquid nitrogen is known, duration for complete evaporation of liquid nitrogen can be calculated.

2.9 CALCULATION FOR REQUIRED VACUUM PRESSURE (R.F.Baron, 1985)
Vacuum Pressure required between inner and outer vessel for the insulation can be find out from the equation,

$Q = G\, P\, A_1 (T_1 – T_2)$

Where, $G = \{[Υ + 1 / Υ – 1] / [gc\, R / 8\, π\, T]^{1/2)}\} \times F_a$

Where Y = specific heat ratio = 1.4

$g_c = 1$

R = specific gas constant = 287 J/kg K

T = Ambient temperature = 300 K

Fa = Accommodation coefficient = 0.9

P= require vacuum pressure

A_1 = surface area for inner vessel

Putting all the required values we can get G and we have Q, therefore we can get the required vacuum pressure between two vessels.

2.10 COLD FINGER (COPPER ROD) (C.A.Bailey & B.A.Hands, 1986)
It is required to transfer cold temperature from inner vessel to the base of sample holder. Therefore, copper rod of suitable length is used. Its lower portion is dipped in to Liquid Nitrogen. Reason to use copper rod as cold finger concept is that it has high thermal conductivity. The governing equation for heat transfer is

$Q = - K\, A\, (T_1 – T_2) / ΔX$

where K = thermal conductivity of copper = 385 W / mK

A = cross sectional area of copper rod

ΔX = length of copper rod

We can optimize the dimensions of cold finger by changing diameter or the length of copper rod.

PROPOSED ATTACHMENT:

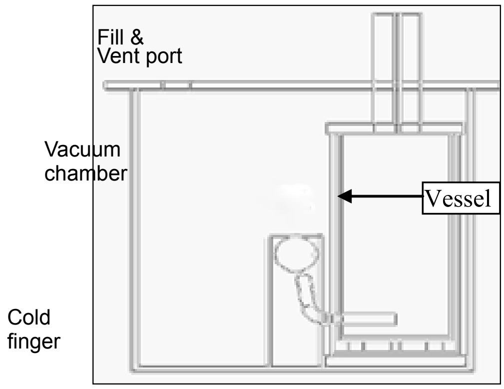

Figure 2 Attachment showing different components

2.10 DESIGN OF VACUUM CHAMBER AND GLASS WINDOW:-
At cryogenic temperatures there arises a problem of condensation of moisture over the surface of the sample as well as on the outer side of LN_2 vessel. However, if both, the sample and the LN_2 vessel are placed in an evacuated chamber than this problem can be eliminated.

According to lame's equation thickness of vessel, t = ri

$$\left[\sqrt{\frac{\sigma t + p}{\sigma t - p}} - 1\right]$$

$= 121*1.97*10^{-4}$

= 0.0119 mm

We have selected thickness of 3 mm which is far greater than required thickness .

Calculation of window thickness:-

$h / d = 1.06\, (P / σ_{ys})^{0.5}$

P = Pressure difference = 0.99 bar

h = thickness

d= window diameter = 0.016 m

$σ_{ys}$ = 68 bar

putting these values we get, h=2 mm

2.11 MANUFACTURING
CONSIDERATIONS (W.J.Tallis, 1986)

Joining Techniques
Joints are the main source of leaks in vacuum systems.
The attachment should be such that, one must be able to utilize the U-V visible spectrophotometer in ambient condition and in cryogenic condition as and when required. Hence, the attachment must be designed taking into consideration the flexibility and portability of operation.

Welding
Tungsten Inert Gas (TIG) welding is most commonly used to weld thin sections of stainless steel. The process grants the operator greater control over the weld than competing processes allowing for stronger, higher quality welds.

Copper Brazing
Copper brazing is preferable to join copper rod as cold finger with inner and outer vessels of SS 304.

CONCLUSION:
This work is a modest attempt of designing a cryocooling attachment for optical spectroscopy. Points which were given due attention, are as follows:

1. The whole system must be free from any vibration and magnetic interference

2. The weight of the system must be as small as possible, to avoid inertia and gravity effects.

3. As the spectrometer chamber is very small, low cooling power is prime requirement.

4. The frosting on the sample and heat losses from the sample cannot be ignored.

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