TECHNICAL SPECIFICATIONS

for

“INSULATION RUBBER MANUFACTURING,
SUPPLY, LINING & ASSOCIATED WORKS
AT SDSC SHAR”
1 ROCASIN RUBBER PROCESSING

1.1 Introduction

ROCASIN is a rubber compound based on the copolymer of Acrylonitrile and polybutadiene known as NBR as per ASTM code. Its formulation contains heat resistant silica filler, cross linking chemicals, plasticizer and other special additives to render ROCASIN as a good insulation rubber compatible with different hardwares. With high tensile strength and strain capability and excellent thermal resistance properties, it finds application in many areas. This ROCASIN rubber was specially developed by ISRO for specific applications and hence the details given here are confidential.

The ROCASIN rubber manufacturing starts with the compounding of the chemicals as specified in the composition and sheeting by extrusion in the unvulcanized condition.

This document gives the processing details of ROCASIN Rubber. The contractor shall have the sufficient infrastructure and the storage facility for both the raw materials and finished products at their factory. Rubber compounding, extruding machinery to manufacture the ROCASIN rubber, meeting the ISRO specifications, shall be available with sufficient manpower to operate in shifts. This product is a white line rubber and hence the machinery should not be used for any other color lines.

1.2 Composition

ROCASIN rubber composition

- NBR - Perbunan NT/3445 (Medium high acrylonitrile)
- Ultrasil VN-3
- Crystex-N
- Dioctyl Phthalate
- Zinc Oxide
- Stearic acid
- Coumarone Indane resin / Capolyte CP-70
- Benzothiazyl disulphide (MBTS)
- Tetramethyl Thiuram Disulphide (TMTD)
- Diethylene Glycol

Raw material specifications are given in Annexure – 4
1.3 Procurement of Raw Materials

The raw materials are available from primary rubber and rubber chemicals manufacturers and their dealers located all over India – especially in the principal industrial centers.

For any change of source in raw materials, clearance is to be obtained from the Purchaser. For accepting the new source, experiment is to be carried out and the data is to be generated. In case of NBR polymer, any change in source/grade (or) introduction of new varieties / brand names shall be done only after obtaining clearance from the Purchaser. The Purchaser’s clearance will be based on evaluation and assessment of mechanical, thermal and interface properties by compounding number of small batches with the chosen NBR polymer.

**Recommended raw materials sources:**

<table>
<thead>
<tr>
<th>MATERIAL</th>
<th>BRAND NAME</th>
<th>MANUFACTURER</th>
</tr>
</thead>
<tbody>
<tr>
<td>NBR Polymer (Medium high acrylonitrilo)</td>
<td>Perbunan NT 3445</td>
<td>M/s. Lanxess, Germany</td>
</tr>
<tr>
<td>Ultrasil VN-3</td>
<td>Ultrasil VN-3</td>
<td>M/s. Insilco, India</td>
</tr>
<tr>
<td>Crystex N sulfur</td>
<td>Crystex N (DS-90 grade)</td>
<td>M/s. Oriental carbon &amp; chemicals</td>
</tr>
<tr>
<td>Dioctyl Phthalate</td>
<td>---</td>
<td>M/s. Indo-Nippon, Bombay</td>
</tr>
<tr>
<td>Zinc Oxide</td>
<td>---</td>
<td>M/s. AVR Zinc products,Pvt. Ltd.</td>
</tr>
<tr>
<td>Stearic acid</td>
<td>Hytitre grade</td>
<td>M/s. Godrej Soaps Ltd., Bombay</td>
</tr>
<tr>
<td>Coumarone Indane resin CP-70</td>
<td>---</td>
<td>M/s. Camphor &amp; Allied Products, Madras</td>
</tr>
<tr>
<td>Mercapto Benzothiazyl disulphide (MBTS)</td>
<td>Vulcacit DM/C Thiofide</td>
<td>M/s. Bayer, India</td>
</tr>
<tr>
<td>Tetramethyl thiuram disulphide (TMTD)</td>
<td>Vulcacitthiuram</td>
<td>M/s. Bayer, India</td>
</tr>
<tr>
<td>Diethyleneglycol</td>
<td>---</td>
<td>M/s. Madras chemical suppliers, Chennai.</td>
</tr>
</tbody>
</table>

1.4 Rubber Processing

ROCASIN rubber shall be manufactured in continuous sheets of 1mm and or 2mm thick as required using a two roll mixing mill and an EXTRUDER. The below process steps may be tailored to suit the infrastructure available with the vendor for achieving the desired properties of ROCASIN rubber with the prior approval of the purchaser.
1.4.1 Premixing

This involves the thorough mixing of all ingredients with the Polymer except the accelerators. A two-roll rubber mixing mill is used for this process. For mixing a ROCASIN batch size of 10 Kgs. usually a mill of size 350 x 900 mm. is required. Before start of the mixing operation, the mixing mill shall be thoroughly cleaned. All the ingredients required for the batch except NBR should be screened to remove any foreign bodies and weighed out accurately. Weighments of all the ingredients are to be carryout precisely in highly accurate balances, which are periodically calibrated. The weighed polymer is taken on the mixing mill (with a close nip) for mastication. During mastication the slabs of polymer are broken down and reduced to a sticky band of sheet holding on to the slower roll. Incorporation of Zinc oxide and stearic acid is carried out next.

*Cumarone Indane resin / Capolyte CP-70 is added, followed by the addition of Ultrasil VN-3. After completing all additions, mixing is continued to get an uniform dispersion. The band is frequently parted off from the mill using scrapper blades and returned to the nip. Fallouts on the pan are swept and reincorporated. After completing the dispersion the compound is released from the mill as a continuous sheeting of about 4 to 5 mm. thick. This thick sheet from each compounded batch is kept under ambient the conditions for de-aeration/cooling for a minimum duration of 12 hours.

1.4.2 Final Mixing & Extrusion

The compound in the form of a blank, corrected to required weight, is once again taken up on the mixing mill and is warmed up by mastication. After the mix has become sufficiently plastic, the accelerators are added and the mixing is continued to ensure uniform dispersion. Extreme care should be taken to ensure thorough dispersion. Fallen materials should be swept and added. At the end of this final mixing the gap between the roller are suitably adjusted to draw a coarse band of 5mm thick. This coarse band is fed directly into the extruder and sheets of 1 or 2mm uniform thickness are produced. The sheets are rolled up in clean cloth.

1.4.3 Storage of Rubber Rolls

Finished rubber rolls are to be stored in an air-conditioned area, till they are dispatched to SDSC SHAR for lining works. The temperatures are to be monitored regularly.

1.4.4 The brief description of the rubber processing is given above. The party has to submit detailed manufacturing process operations and quality assurance procedures to be followed, along with the quote.

The party has to carry out qualification trials following the details given in Phase 1 and Phase 2 trials to finalize the manufacturing process and batch size to suite the available machinery and obtain the approval from the purchaser before use.
1.5. Rubber Manufacturing Process Qualification Trials:

1.5.1. PHASE-1 Trials:

a. Supply of approx. 50 Kg of Extruded ROCASIN rubber sheet processed using the infrastructure identified for ROCASIN rubber manufacture as per the adapted procedure.

b. Acceptance criteria for the above sheet:
   - Thickness: 2 ± 0.2mm and 1 ± 0.1mm sheets.
   - Appearance: Free from surface defects and discoloration.
   - Free from air inclusions and blisters

c. Sample sheets drawn from the above rolls will be tested for the Mechanical properties, Metal to rubber peel bond strength, Twaron to rubber Tensile & peel bond strength, Thermal properties and Rheological properties mentioned in 2.1 to 2.5 of the Rubber Specifications part of technical document.

1.5.2. PHASE-2 Trials:

Evaluation of the interfacial bond properties and Subscale motor test following Quality acceptance plan laid down by ISRO are to be carried out after order confirmation.

   a. Evaluation of Interfacial bond properties: Propellant to rubber, inhibition to rubber, will be evaluated
   b. Subscale motor test for evaluating the thermal performance.
   c. Evaluation of shelf life of ROCASIN rubber.

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After manufacturing, the Extruded ROCASIN rubber sheet will be delivered at SDSC SHAR. The following inspection procedures will be followed for the acceptance.

2.1 Visual & Dimensional Inspection

The extruded ROCASIN rubber shall be supplied in rolls of 20 to 25 meters with a nominal width of 1 meter and it shall have a smooth surface, free from pores, bubbles, inclusions and other surface defects. The thickness shall be 1 or 2mm with a tolerance of ± 10%. The thickness measurement is done on number of random locations across the width and length of the sheet.

2.2 Mechanical & Physical Properties

For this purpose, from each roll a sample will be drawn, identified by a Roll No, vulcanized and properties evaluated. Samples are vulcanized in a hot air oven at 120° C
for 4 hrs and then sent to Mechanical property evaluation. This testing will be carried out by Purchaser and shall meet the following specifications.

<table>
<thead>
<tr>
<th>Tensile Strength</th>
<th>kg/sq.cm</th>
<th>100 minimum</th>
</tr>
</thead>
<tbody>
<tr>
<td>Elongation (%)</td>
<td></td>
<td>600 minimum</td>
</tr>
<tr>
<td>Hardness Shore -A</td>
<td></td>
<td>60 - 75</td>
</tr>
<tr>
<td>Density gm /cc</td>
<td></td>
<td>1.19 ± 0.02</td>
</tr>
</tbody>
</table>

2.3. Thermal Properties

The party has to prepare samples for thermal property evaluation and deliver for every lot of Rubber supply from the first roll, middle roll and the last roll of the rubber quantity being supplied. The rubber lot shall be used for Rubber lining only after obtaining the results and acceptance. These tests are required for perbunan NT 3445 lot qualification and subsequently once in six months on storage. In addition, thermal properties are to be evaluated for change of source/lot of NT 3445 & Ultrasil VN3 Silica. These tests are carried out by Purchaser and shall meet the following specifications.

<table>
<thead>
<tr>
<th>Thermal conductivity (cal/cm sec.ºC)</th>
<th>: 6.5 x 10⁻⁴ maximum</th>
</tr>
</thead>
<tbody>
<tr>
<td>Specific heat (cal/gm.ºC)</td>
<td>: 0.35 minimum</td>
</tr>
<tr>
<td>Coefficient of linear Expansion (mm/mm.ºC)</td>
<td>: 3 x 10⁻⁴ maximum</td>
</tr>
<tr>
<td>Erosion rate (300 W/sq.cm)</td>
<td>: 0.2 mm/sec Maximum</td>
</tr>
</tbody>
</table>

2.4 Acrylonitrile Content

In addition to the physical properties check, chemical analysis of ROCASIN is to be done by the party to determine the acrylonitrile content. The specified acrylonitrile content of ROCASIN rubber is 16-19%. This is to be done once in six months for accepted lot of rubber.

2.5 Rheological properties:
Rheological properties shall be evaluated by the party for the rubber whenever required.
Major steps in insulation lining and associated activities operation of the hardware with Rocasin sheet is described below:

3.1 HARDWARE SURFACE PREPARATION OPERATIONS

3.1.1 Preliminary Degreasing

The inner surface of the hardware is cleaned thoroughly with TCE solvent to remove lacquer, oil, grease, dust etc. This cleaning is done manually by wiping the surface with TCE soaked cloth. After visual, dimensional inspection and clearance, the machined metal assembly interfaces of the hardware, that is not to be blast cleaned, is masked with 2 mm thick unvulcanized ROCASIN rubber as per the detailed masking procedure. In case of specific requirement additional 2 mm thick pads and cello/Teflon tapes are also used.

3.1.2 Blast Cleaning

Inner surface of the hardware is blast cleaned using chilled iron grits of size SAE-G40 at a pressure of about 50 Psig. During blasting, extreme care is to be taken to ensure that no over blasting is done anywhere, as it would erode away the hardware material and reduce the thickness of shell. (Alternatively manual sand blasting with river sand at a pressure of about 70 Psig can also be carried out in case of requirement). Condition of blast cleaned surface is visually checked for cleanliness (removal of rust/scale) for acceptance. If blast cleaned surface is not satisfactory, the blast cleaning operation has to be carried out after scrapping left over paint, again till such time to obtain SA 2 ½ Grade surface cleanliness. Any surface defects in the hardware are to be reported and obtain clearance before taking up for other operations. The machined assembly interfaces of the hardware not to be blast cleaned are to be masked with un-vulcanized rubber and Teflon/cecko tapes.

3.1.3 Final Degreasing

After blast cleaning the hardware surface is swept free off grits and dust by blowing compressed air and wiping manually. The masking over the machined interfaces are removed where ever required. Blast cleaned surface is washed clean using degreasing machine with TCE solvent, thoroughly to remove grit dust. After washing with the solvent the chamber inner surface is visually examined for cleanliness. The surface free from rust, mill scale and dust only will be cleared for further processing.

3.1.4 Primer Application

After completion of final degreasing operation, the cleaned surface is coated with Chemlok 205/Unilok 205 by brushing. The initial weight of the chamber, before rubber lining operations, is taken and recorded at this stage. The specifications of chemlok –205 is given below:

- Viscosity at 25°C Cps : 85 – 165
- Solids contents % : 20 – 26

The metal machined interfaces shall be protected from rust using rubber solution and Teflon sheet.
5 to 10 Nos of metal samples (approx) of size 55mm x 25mm x 4 mm are also blast cleaned, solvent cleaned and applied with primer along with the hardware processing for the preparation of interfacial bond strength evaluation specimens to represent the conditions of the hardware being processed.

3.2 LINING OPERATIONS:

3.2.1 Rubber Solution Preparation

ROCASIN rubber from qualified lot is cut into small pieces. This material is taken in a clean plastic container (with lid). To this, Binary solvent is added and allowed to soak overnight. Rubber solution is obtained by homogenizing soaked rubber pieces using a mechanical stirrer. Identification batch No., date of preparation etc., are recorded in a logbook.

Consistency of rubber solution for brush application is maintained by adding binary solvent, if needed. The prepared batch of rubber solution should be used within the shelf life expiry period (4 weeks from the date of accelerator addition) of the rubber adhesive roll identified for this purpose.

3.2.2 Rubber Solution Application

Rubber solution applied over the primer-coated surface in two coats allowing a drying period of 2 hours in between the first and second coat. One hour drying period is allowed after second coat of solution application prior to start of rubber lining work.

3.2.3 Lining Process

Before commencing rubber lining works, at the Tang end of hardware suitable support pad is to be inserted into the crevices of the hardware to act as a support for the overhanging portion of the insulation. The support pad is prepared by bonding 2mm thick pre-vulcanized rubber sheets to the required dimensions. Additionally, a film of Teflon Coated Fiber Glass tape is bonded to prevent bonding of ROCASIN lining to this support.

Unvulcanised ROCASIN sheets of 1 & 2 mm thick are employed for insulation lining. The final insulation lining drawing will be provided by the Purchaser at the time of commencement of work. Each of the ROCASIN rubber sheets is thoroughly cleaned with TCE (bonding surface) and brushed with rubber solution before bonding to the chamber. The bonded sheet is then rolled using hand rollers to expel the entrapped air. During lining work, operators should wear caps, socks etc., so as not to contaminate the prepared surface.

After completing base layer of insulation lining, ultrasonic test is conducted by the Purchaser to detect the de-bonds between insulation and hardware. After rectification of these defects, Spark Testing is carried out by the Contractor to detect pinholes if any. Necessary fillings are carried out and re-tested to ensure absence of pinholes.

Surface prepared representative samples are lined and preserved for vulcanization.
Subsequently, additional layers of rubber lining are carried out as per lay up drawings, to build up necessary thickness of insulation. The overlap joints are staggered from layer to layer to avoid excessive thickness build up over the joints.

After completion of insulation lining marking of the bonded and un-bonded regions of the loose flap on the main insulation has to be carried out. A separation film (Teflon Coated Fiber glass cloth without adhesive) is pasted wherever un-bonded loose flap is required. This un-bonded loose flap is retained close to the contour of the main insulation by bonding it to the separation film with rubber solution. Loose flap lining is carried out with a 2mm thick ROCASIN sheet.

3.2.4 Composite Lining on End Domes

Composite lining comprises of lining a layer of carbon cloth over the previously laid ROCASIN rubber layer. Alternate ROCASIN layer and carbon cloth layer carry out this work till the required number of composite layers are completed. After carrying out built-up thickness and axial measurements, a covering layer of 2 mm thick (top layer) ROCASIN in lined to complete the work. Composite lining work involves the following steps:

(a) Preparation of carbon cloth:

From identified carbon cloth rolls (Dept. Supply) convenient lengths are cut (10-15 meters) and soaked in TCE solvent in a plastic bucket for an hour. The soaked cloth is taken out and left to dry in ambient conditions.

(b) Application of rubber adhesive:

On both the sides of the dried carbon cloth, a coat of adhesive is applied and allowed to dry. After drying the carbon cloth is rolled in a polyethylene sheet and stored for usage whenever required.

(c) Preparation for lining of carbon cloth:

The rubber adhesive coated carbon cloth is cut to size and one more coat of rubber solution is applied on to the side to be bonded. Rubber solution is allowed to dry before using the sized carbon cloth.

(d) Lining of carbon cloth over ROCASIN:

The sector of ROCASIN rubber sheet to be bonded with prepared carbon cloth is freshened with TCE and a coat of rubber solution is applied. After the evaporation of the solvent from the adhesive, the sized carbon cloth is placed over the ROCASIN rubber, adjusted to avoid folding, and bonded using hand rollers. After completing rolling, the carbon cloth surface is inspected for entrapped volatiles and released before re-rolling to effect bonding. If required re-work is to be carryout Representative samples of Rocasin& carbon of relevant carbon and rubber used lot to be made and preserved for vulcanization.
3.3 VACUUM BAGGING OF THE RUBBER LINED HARDWARES

The end portion of Hardwares and Bucket flange where the lining thickness is large needs to be vacuum bagged prior to insulation vulcanisation. Vacuum bagging consists of the following works.

(a) Laying of Release Ply material on Insulation at both ends with Fevicol (tack bonding)

(b) “Bridge Material” (Cured ROCASIN sheets cut to required size) bonding in required number of layers (2 to 3 Nos) with Fevicol at both ends.

(c) Tack bonding of “Breather Material” with Fevicol at both ends.

(d) Paint removal for 20mm width (by scrapping) on external surface of Hardware for vacuum putty bonding at both ends.

(e) Preparing “Nylon Film” with required number of joints for enclosing “Breather Material” at both ends. (Required Number of Nipples are to be kept in position with vacuum putty)

(f) Vacuum Putty bonding at two ends of “Nylon Film” at both ends.

(g) After completion of vulcanization, all these “Bag” materials are to be removed. Bridge material to be cleaned with solvent so that they can be used for next segment/motor.

(h) Vacuum putty on Insulation surface to be thoroughly removed with solvent.

(i) External surfaces where paint was removed to be repainted with Paint.

3.4 Vulcanization of Insulation lining of Rocket hardwares in Hot Air Autoclave: The Insulation lined segment hardware with vacuum bagging at the ends is loaded inside the Hot Air Autoclave. The hardware is subjected to Temperature of 120°C and pressure of 5 Bar inside the autoclave in a specific pattern in the time cycle which last for 10 to 14 hours. The thick insulation at the hardware ends are subjected to vacuum by connecting it to the vacuum port. Vulcanization is done in Automatic mode where all the subsystems/equipments involved are operated and controlled by PLC/Controller. The above process involves the following:

(i) Handling and loading of the Rocket hardware by using lifting Tackles and belts with the help of Crane.

(ii) Operation of Autoclave and data logging to complete the vulcanization cycle which also includes operating compressors, vacuum pumps, cooling system & Tower.

(iii) Unloading of the Rocket hardware from autoclave after completion of vulcanization by using lifting Tackles and belts with the help of Crane.

Representative samples prepared along with hardware processing are to be vulcanized along with hardwares. 1 to 2 ROCASIN sheets of 1.5 x 1 sqm are also vulcanized for TBS, Peel & IC sample preparation.
3.5 POST VULCANIZATION OPERATIONS:

After vulcanization, the separation film inserted in between the loose flap and the main insulation is to be removed. Hardware assembly interfaces surfaces are to be cleared of rubber adhesive. Tap testing is to be carried out by the Contractor to check for any defects. At this point the final weight of the hardware with insulation is taken and recorded. The machined assembly interfaces of the hardware shall be protected using rust protective coating.

3.6 ABRASION OF THE VULCANISED INSULATION SURFACE

Insulation abrasion is done to remove superficial contaminants from the insulation surface before preheating operation. Initially the abrading flap wheels shall be qualified by abrading on a sample sheet out side to ensure that the wheels leave no contamination on the surface being abraded. During abrasion work, the operator should make use of polythene sheet/kora cloth to avoid contaminating the insulation surface. The workers shall use self protective equipment like leather gloves, goggles and dust mask during operation, the abrasion should be uniform and should be carefully done to avoid excessive material removal.

While abrading over the unbonded loose flaps, the operator shall be properly instructed to avoid cuts/tears on the loose flaps. Inadvertent damage caused to the loose flap by way of excessive thinning/local holes if any be shall repaired. The abrasion operation shall be carried out sector by sector for covering the entire surface. TBS, PEEL and IC samples are also abraded.

After abrasion operation the segment should be thoroughly cleaned by coir brush to remove dust. After completion of abrasion, the surface shall be inspected for certification of operation. Wherever necessary, touch up work on un-abraded insulation surface is to be done to make the insulation surface uniformly abraded before final acceptance.

3.7 SPACER, BELLOW BONDING & LOOSE FLAP EXTENSION

Solvent cleaning of insulation surface has to be carried out in between the loose flap and main insulation on the hardware on ends. Spacer bonding on both the ends, Bellow preparation and bonding, stitching on the other end as per the relevant document. At one end of the segment/case is to be extended with un-vulcanized rocasin rubber as per requirement. The said spacer and bellows are the small pieces prepared by Rocasin at site and having weight of less than 500 grams.

Loose Flap venting: Loose flap venting is to be incorporated as per the approved schemes. 2 to 4 nos of vent channels are incorporated at both the ends. These venting provisions are obtained by punching holes on bellow and bonding strips of breather material between Loose flap and insulation or Hardware.
3.8 SOLVENT CLEANING

Abraded insulation surface is cleaned with solvent to ensure no contamination is present on Insulation surface. TCE is used as solvent and cleaning is done manually at ambient conditions. TCE cleaning work content is given below.

(a) Initially entire insulation surface is thoroughly cleaned with coir brush for ensuring Insulation dust generated during Abrading is totally removed. This dust cleaning operation is to be done twice.

(b) Mill cloth in required quantity is to be de-starched by soaking in water and subsequently dried for use in the cleaning operation. Mill cloth is to be cut to required size and number of pieces.

(c) Required quantity TCE is taken in a plastic bucket and kept near the segment/motor (outside). Mill cloth pieces dipped in TCE taken out, excess TCE squeezed out and then the cloth is used for Insulation cleaning. Entire surface is to be cleaned by Mill cloth pieces with TCE. After completion of one round of cleaning, 2\textsuperscript{nd} round of cleaning on the entire surface is to be completed.

Thoroughness of TCE cleaning is checked for acceptance. Solvent cleaning is to be repeated, if contamination is observed. Preparation of kora cloth covers for closing the segment ends and loading in the Oven/Autoclave.

3.9 PREHEATING OF INSULATION LINED ROCKET HARDWARE

Rocket Hardware is loaded inside the Hot Air Autoclave. The Rocket hardware is subjected to Temperature of 105°C for the duration and total process lasts for 26 Hours. Preheating is done in Automatic mode where all the subsystems are operated and controlled by PLC/Controller. The above process involves the following:

The above process involves the following:

(i) Handling and loading of the Rocket hardware by using lifting Tackles and belts with the help of Crane.

(ii) Operation of Autoclave and data logging to complete the Preheating cycle which also includes operating cooling system & Tower.

(iii) Unloading of the Rocket hardware from autoclave after completion of Preheating by using lifting Tackles and belts with the help of Crane.

3.10 RESIN LINING OF ROCKET HARDWARE SEGMENTS

Resin Liner is spray coated on the inner surface of the insulation lined rocket hardware to achieve a nominal thickness of 100 microns using Resin Lining Machine. Major activities followed in Resin Lining are as follows:
Weighment of Resin Liner premix and curator by electronic weighing balance:
Premix: Approximate 5 Kg to 15 Kg.
Curator: Approximate 0.5 Kg to 2 Kg

De-gasification of TDI added Resin Liner in a Vacuum chamber of size 100 liters by operating vacuum pump for a period of 30 minutes/batch of size of 5 Kg(approx).

Transferring metered quantity of solvent Di-Chloromethane in Flask from bottles of 2.5 litres capacity. Quantity of Solvent DCM being used is in range of 4 liters to 15 liters based on the batch size and stirred for about 5 minutes.

Loading of the Rocket hardware on Resin lining roller stands by using lifting Tackles and belts with the help of Crane.

Masking of the rocket hardware ends using polythene sheets/Teflon sheets to prevent the liner being coated on the hardware machined joint interfaces. Double sided adhesive tapes of size 1 inch/2 inch are used for the bonding the polythene sheets for a width of about 500mm.

Bonding of Rubber samples on the inner surface of the End Rings using finical. The size and No of Rubber samples to be bonded is given below.

<table>
<thead>
<tr>
<th>Type</th>
<th>Size</th>
<th>No.</th>
</tr>
</thead>
<tbody>
<tr>
<td>TBS samples</td>
<td>95mm x 125mm</td>
<td>20 Nos. (max)</td>
</tr>
<tr>
<td>Peel Samples</td>
<td>335mm x 150mm</td>
<td>10 Nos. (max)</td>
</tr>
<tr>
<td>IC Samples</td>
<td>145mm dia, 475mm X 155mm</td>
<td>5 Nos. (max)</td>
</tr>
</tbody>
</table>

Operation of Resin Lining Machine and data logging to complete the Resin Lining. This involves synchronized rotation of rocket hardware on the roller stand and longitudinal travel of boom carrying spray gun to achieve uniform liner spray coating. Lance is swiveled to orient the spray gun in the desired location to cover domed ends of Head End and NE segments. Measured quantity of liner is delivered by metering pump and atomized by nitrogen supplied at 3 bar(approx). The above operations are done in Automatic mode where all the subsystems are operated and controlled by PLC/Controller.

Removing the above liner coated samples are preserved in the trays. Following days, these samples are bonded on the TBS Blocks of size 95mmx125mmx25mm and Peel drums of dia 107mm x 150mm long.

Cleaning of liner metering pumps, liner spray gun and liner feed line using solvent TCE.

Closing of both the ends of the rocket segments after completion of liner coating by using polyethylene sheet using double sided adhesive tapes.

Purging the above covered inner volume of lined hardware by nitrogen gas

Unloading of the Rocket hardware from resin lining roller stand by using lifting Tackles and belts with the help of Crane.

Tilting of the Rocket hardware to vertical orientation by “A - Frame Tilting Fixture” and crane.
Lifting of the rocket hardware and Positioning on the trailer.

Readiness checks for resin lining operations are to be carried out before the resin lining operation. Major activities involved in readiness are as follows.
(a) Cleaning of liner metering pumps and liner spray gun.
(b) Laying and cleaning of nitrogen and liner feed line.
(c) Assembling of Resin metering pumps, spray gun, gauges, nitrogen cylinder, etc.
(d) Checking the functioning of roller stand, boom and lance.
(e) Trial run of the Resin lining machine in auto mode using PLC.
(f) Readiness of liner vacuum application and degasification unit.
### Hardware Lining Area Details

<table>
<thead>
<tr>
<th>Hardware</th>
<th>Base layer area (m²) 2mm thick</th>
<th>Subsequent layer area (m²) 1mm thick</th>
<th>Subsequent layer area (m²) 2mm thick</th>
<th>Vacuum bagging</th>
<th>Abrading</th>
<th>Solvent cleaning, Spacer, Bellow bonding &amp; LF Extension</th>
</tr>
</thead>
<tbody>
<tr>
<td>Hardware – 1 (S200 HES)</td>
<td>30</td>
<td>30</td>
<td>275</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Hardware – 2 (S200 MS)</td>
<td>80</td>
<td>21</td>
<td>176</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Hardware – 3 (S200 NES)</td>
<td>82</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>Rocasin: 690 Carbon Cloth: 200</td>
</tr>
<tr>
<td>Hardware – 4 (PSOMXL HES)</td>
<td>13</td>
<td></td>
<td>17</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Hardware-5 (PSOMXL MS)</td>
<td>14</td>
<td></td>
<td>17</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Hardware-6 (PSOMXL NES)</td>
<td>14</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>Rocasin: 125 Carbon Cloth: 22</td>
</tr>
<tr>
<td>Hardware – 7 (S200 BFL)</td>
<td>3</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>Rocasin: 146 Carbon Composite: 100</td>
</tr>
<tr>
<td>Hardware 8 (S139 MS)</td>
<td>30</td>
<td>25</td>
<td>190</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Hardware 9 (S139 NES)</td>
<td>30</td>
<td>23</td>
<td></td>
<td></td>
<td></td>
<td>Rocasin: 280 Carbon Cloth: 145</td>
</tr>
<tr>
<td>Hardware 10 (S139 HES)</td>
<td>30</td>
<td></td>
<td>380</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Miscellaneous works (End support pads, Vacuum bagging support pads &amp; LF Extension works)</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

**Note:** These figures are indicative for the type of job involved. Actual lay-up drawings will be provided to the contractor prior to taking up of lining operations. In case of necessity to revise the drawings, approved scheme will be furnished during the course of rubber lining. The lay-up scheme followed during the execution will be the reference for computing actual lining area for each component and shall be followed.

CC: Carbon cloth composite lining
4. QUALITY CONTROL & INSPECTION

Quality control and Inspection action comes in at five stages as given below:

- Raw material procurement stage
- Rubber sheet manufacturing stage
- Rubber sheets acceptance stage
- Hardware surface preparation stage
- Insulation lining stage.

4.1 All the raw materials used for processing of rubber sheets (ROCASIN) shall be analyzed and tested and only those meeting laid down specifications as detailed in Annexure-4 shall be used by the contractor for compounding. The accepted raw materials shall be separately identified and stored. The analysis/test certificates issued by Government approved laboratories shall be provided to the Purchaser.

4.2 Rubber rolls after receipt at SDSC SHAR shall be tested by Purchaser for mechanical properties and other acceptance parameters as specified in section 2. For this purpose adequate number of samples will be drawn from each roll and tested as per the standard procedures at SDSC SHAR. Only those rolls which meet the specification will be accepted for adhesive preparation/lining works.

In addition, the contractor shall also qualify the rolls, using his facilities, prior to dispatch to ensure that rubber rolls meeting specifications only are sent to work site.

The contractor shall provide all facilities for the Purchaser’s inspection personnel for carrying the stage inspection as well as product inspection. The Purchaser’s engineers shall have the liberty to draw the samples of the raw materials/finished products at any stage of manufacture for evaluation/testing. The Purchaser shall test the rubber rolls supplied by the contractor and on testing if the sheets are not in accordance with the specifications and other acceptance parameters, the same shall be rejected.

4.3 Visual and dimensional inspection of the rubber sheets shall be carried out prior to acceptance. The surface shall be smooth and be free from voids, cuts, blisters, blooming, discoloration and contamination.

4.4 All the in-process inspection at manufacture site/at work center should be done by the contractor and certified. However, for the initial few batches surveillance may be done by the concerned personnel of the Purchaser. Mechanical properties of the sheet is to be evaluated for all rolls. Thermal properties are to be evaluated for 1st roll, middle roll and last roll of each lot. Thermal properties of rubber are to be evaluated for every new lot of PERBUNAN polymer or silica for purchaser’s clearance for processing rubber. The same are to be repeated at every 6 months for revalidation. The vendor shall supply moulded samples for thermal properties evaluation as per the specifications provided by the Purchaser for each vendor’s lot as detailed above.

4.5 The hardware surface shall be inspected at various stages as indicated below:

- After preliminary cleaning/degreasing.
- After blast cleaning
4.6 Primer Chemlok 205 shall be used only after clearance/qualification by Purchaser (adequate primer tins are to be kept in stock at SDSC SHAR to avoid delay in carrying out rubber lining works).

4.7 Ultrasonic testing shall be carried out by Purchaser after completion of base layer lining to detect air gaps, if any. Defective areas located shall be corrected by the contractor immediately, which would again be reconfirmed for defect free bonding by ultrasonic testing. The contractor shall carry out Spark testing in presence of Purchaser’s Engineer for detection & correction of pinholes in the base layer before lining of subsequent layers. Spark testing of rubber sheets where ever is necessary before base layer lay up to be carried out.

4.8 Visual and dimensional inspection shall be carried out by the contractor in the presence of Purchaser representative during different stages of rubber lining.

4.9 Tap testing and visual inspection shall be carried out by the contractor in the presence of Purchaser representative during different stages of rubber lining.

4.10 The process log sheets as required by the Purchaser shall be maintained by the contractor and calibration of the measuring instruments like weighing balances, temperature gauges shall be carried out periodically and the data to be supplied to the Purchaser.

4.11 Process details is to be filled by the contractor in the log formats given by the Purchaser.

4.12 Raw material analysis procedures given by the Purchaser shall be strictly followed. In case any clarifications regarding the analysis procedures are required, the same may be obtained from SPP, SDSC SHAR.

4.13 A government approved test house shall be used for analysis of all the raw materials. Purchaser shall be involved while finalizing the test house.

4.14 All the raw materials used for the processing of rubber shall be identified with a distinct lot no/ batch no.

4.15 Contractor shall maintain the process log in such a way that any processed “rubber lot” shall have the trace back to get the total history of the raw materials used and the process conditions employed. These logs will be periodically checked by the Purchaser and if asked the copy shall be produced.

4.16 Quality on the process and related activities will be carried out by the Purchaser periodically. All the input data for this shall be made available to the quality team.

4.17 Any suggestions made from time to time to improve the quality of the product shall be incorporated in the process by the contractor.

4.18 The analytical report of the raw materials are to provided to SDSC - SHAR before use in rubber manufacturing and shall get clearance from the Purchaser for use.
Raw materials specification – Rubber Processing:

**NBR (Medium high Acrylonitrile) Perbunan NT3445**

- **Physical form**: Slabs, crumbs or biscuits
- **Colour**: Translucent honey coloured
- **Specific gravity @ 30°C**: 0.97 – 1.00
- **Acrylonitrile content**: 30-35%
- **Mooney viscosity**: 45 ± 5

**Ultrasil VN3 (Specific brand)**

- **Physical form**: Fine white powder
- **Specific gravity at 30°C**: 2.0 ± 0.1
- **Silica as SiO₂**: 85% min.
- **Weight loss (105°C, 2 hours)**: 7% max.
- **Weight loss on ignition (800°C, 2 hours)**: 12.5% max.
- **pH of 5% aqueous slurry**: 6.00 ± 0.5

**Crystex Sulphur**

- **Specific gravity at 30°C**: 2 ± 0.1
- **Purity (Assay)**: 90% (min.)
- **Insoluble Sulphur**: 99% (min)
- **Acidity**: 0.15% max.
- **Ash**: 0.15% max.
- **Grade**: Non oil-extended

**Zinc Oxide**

- **Physical form**: White powder
- **Specific gravity @ 30°C**: 5.4 – 6.0
- **Particle size**: 4 microns max.
- **Purity**: 95% min.
- **Grade**: White seal

**Stearic Acid**

- **Physical form**: White wax like solid
- **Specific gravity @ 30°C**: 0.94 – 0.98
- **Melting point**: 68 – 70°C
- **Purity (by carboxy value)**: 98% min.
**Dioctyl Phthalate**

Physical form : Colourless liquid  
Specific gravity @ 30°C : 0.97 – 0.99  
Volatile matter (@105°C, 2 hours) : 0.2% max.  
Saponification value : 288.7 ± 3 mg. KOH/gm.  
Acidity (max.) : 0.5 mg. KOH/gm.  
Moisture content : 0.1% max.

**Cumarone Indane resin**

Physical form : Resin like yellow solid  
Specific gravity @ 30°C : 0.90 – 1.10  
Melting Range : 85 – 95°C

**Capolyte CP 70**

Specific gravity @ 30°C : 0.90 – 1.10  
Melting Range : 65 – 75°C

Note: Cumarone Indane resin (or) Capolyte CP 70 can be used.

**Diethylene Glycol**

Physical form : Colourless liquid  
Boiling point : 245°C  
Specific gravity @ 30°C : 1.11 – 1.12  
Refractive Index @ 30°C : 1.442 – 1.446

**Benzo Thiazyl Disulphide (MBTS)**

Physical form : Yellow white to grey white powder  
Specific gravity at 30°C : 1.3 – 1.4  
Melting point : 174 – 180°C  
Total Sulphur content : 36 – 39%

**Tetramethyl Thiuram Disulphide (TMTD)**

Physical form : Yellowish powder  
Specific gravity @ 30°C : 1.4 – 1.5  
Total sulphur content : 51 – 53%  
Melting point : 140°C (min.)
ANALYSIS METHODS FOR RAW MATERIALS

1  ULTRASIL - VN-3 (Pyrogenic Silica)

1.1  Specific Gravity:

Specific gravity is found out using a specific gravity bottle with water.

Take a clean dry specific gravity bottle and find its weight with stopper accurately. Fill about 1/3rd of its volume with the sample. Put the stopper and find out the weight of the specific gravity bottle with the sample. Then fill the bottle 1/3 with water over the sample. De-aerate for half an hour to remove the air bubbles. Then fill with water. Put the stopper. Wipe out the surface of the specific gravity bottle with blotting paper and find the weight. Now empty the bottle. Clean dry and fill it with distilled water. After putting the stopper without air bubbles, wipe the surface and note the weight with distilled water alone.

\[
\text{Wt. of specific gravity bottle} = W_1 \text{ gm}
\]
\[
\text{Wt. of specific gravity bottle + sample} = W_2 \text{ gm}
\]
\[
\text{Wt. of specific gravity bottle + sample + water} = W_3 \text{ gm}
\]
\[
\text{Wt. of specific gravity bottle + water} = W_4 \text{ gm}
\]

\[
\text{Sp. Gravity} = \frac{(W_2 - W_1)}{(W_4 - W_1) - (W_3 - W_2)}
\]

1.2  Silica as SiO$_2$

Take 1 gm of Ultrasil VN-3 in a platinum crucible. Add 2 – 3 drops of conc. H$_2$SO$_4$, followed by HF (about 1 ml). Continuously heat on hot plate till all the dense fumes of hydrofauro silica acid are expelled. (about 4 hours). Heat the crucible muffle furnace at 800°C for 2 hours and cool in desiccator. Weight the residue left. Find the SiO$_2$ percentage. Subtract the percentage of loss on ignition to get the SiO$_2$ content.

\[
\text{Wt. of platinum crucible} = W_1 \text{ gm}
\]
\[
\text{Wt. of crucible + sample} = W_2 \text{ gm}
\]
Before heating
\[
\text{Wt. of crucible + sample} = W_3 \text{ gm}
\]
After heating
\[
\text{Loss on ignition} = X \%
\]

\[
\text{Silica content ( %)} = \left\{ \frac{(W_3 - W_2)}{(W_2 - W_1)} \right\} \times 100 - X
\]
1.3 **Weight loss at 105°C:**

Weight exactly 10 gms of the material in a crystallization dish which is dried and previously weighed. Heat the dish in an air oven kept at 105°C for 2 hours. Finally cool and weight the dish.

\[
\text{Wt.loss at 105°C (%) = \frac{W1 - W2}{W1 - W} \times 100}
\]

\(W1\) = Wt.of dish + sample before heating
\(W2\) = Wt.of dish + sample after heating

1.4 **Loss on Ignition**

Weight exactly 5 gm of the material in a dry platinum crucible. Keep it in an electric furnace for 2 hours at 800°C. After ignition cool and weigh the crucible.

\[
\text{Loss on ignition (%) = \frac{W1 - W2}{W1 - W} \times 100}
\]

\(W\) = Wt.of empty platinum crucible
\(W1\) = Wt.of crucible + silica before heating
\(W2\) = Wt.of crucible + silica after heating

1.5 **pH of aqueous slurry**

Weight 5 gm of material into 100 ml of previously boiled and cooled distilled water and stir well. Take the pH of the slurry using a calibrated pH meter.

2 **CRYSTEX SULPHUR / DIAMOND SULPHUR (DS 90):**

2.1 **Specific gravity:**

Specific gravity is found out using a specific gravity bottle using alcohol/light liquid paraffin oil. Take a clean dry specific gravity bottle and find its weight with stopper accurately. Fill about 1.3 rd of its volume with the sample. Put the stopper and find the weight of the specific gravity bottle with the sample. Then fill the bottle 1/3rd with ethyl alcohol over the sample. De-aerate for half an hour to remove the air bubbles. Then fill with ethyl alcohol. Put the stopper, wipe out the surface of the specific gravity bottle with blotting paper and find the weight. Now empty the bottle, clean and fill it with ethyl alcohol alone and find out its weight after putting stopper and wiping out the surface with blotting paper. Finally empty the bottle, clean and dry and fill it with distilled water. After putting the stopper without air bubbles, wipe the surface and note the weight with distilled water alone.
**Calculation**

Wt. of empty Sp. gravity bottle = W1 gm
Wt. of Sp. gravity bottle + sample = W2 gm
Wt. of Sp. gravity bottle + sample + ethyl alcohol = W3 gm
Wt. of Sp. gravity bottle + ethyl alcohol alone = W4 gm
Wt. of Sp. gravity bottle + water = W5 gm

\[
\text{Sp. Gravity} = \frac{(W2 - W1) \times (W4 - W1)}{(W4 - W1) - (W3 - W2) \times (W5 - W1)}
\]

2.2 **Insoluble Sulphur**

2 gms of the material is stirred with 50 ml carbon disulphide and filtered through a G-4 sintered crucible. Wash the residue with LR grade CS2 four times and weigh the residue.

Wt. of sample = W1 gm
Wt. of the residue = W2 gm

\[
\text{Insoluble sulphur ( %)} = \frac{W2}{W1} \times 100
\]

2.3 **Ash:**

5 gm of the material is weighed in a silica crucible and slowly burned. Heat the crucible at 800 – 900°C muffle furnace for at least one hour. Cool in desiccators and weigh.

Wt. of silica crucible = W1 gm
Wt. of crucible + sample before heating = W2 gm
Wt. of crucible + sample after heating = W3 gm

\[
\text{Ash ( %)} = \frac{(W3 - W1)}{(W2 - W1)} \times 100
\]

2.4 **Purity:**

Refer the procedure for TMTDS. Calculate purity by Sulphur.
3. **ZINC OXIDE**

3.1 **Specific Gravity**

Specific gravity is found out using specific gravity bottle with water.

Take a clean dry specific gravity bottle and find its weight with stopper accurately. Fill about 1/3rd of the volume with the zinc oxide. Put the stopper and find out the weight of the specific gravity bottle with the sample. Then fill the bottle 1/3rd with water over the sample. De-aerate for half an hour to remove the air bubbles. Then fill with water, put the stopper, wipe out the surface. Clean the specific gravity bottle with blotting paper and find the weight. Now empty the bottle, clean dry and fill it with distilled water. After putting the stopper without air bubbles, wipe the surface and note the weight with distilled water alone.

**Calculation**

\[
\text{Wt. of empty Sp. gravity bottle} = W_1 \text{ gm} \\
\text{Wt. of Sp. gravity bottle + sample} = W_2 \text{ gm} \\
\text{Wt. of Sp. gravity bottle + sample + water} = W_3 \text{ gm} \\
\text{Wt. of Sp. gravity bottle + water} = W_4 \text{ gm}
\]

\[
\text{Specific Gravity} = \frac{(W_2 - W_1)}{(W_4 - W_1) - (W_3 - W_2)}
\]

3.2 **Particle size**

Particle size is found out using sub-sieve sizer. First of all check up the sub-sieve for its good work condition. Now insert the standard tube in the measuring circuit and standardize the instrument for both ranges up to 20 microns and 20 to 50 microns at 0.75 porosity. Weigh exactly 5-6 gm of the sample of zinc oxide and pour it in to the sample tube provided at the lower part of the porous plate covered with disc of filter paper. Put the second plate at the upper part covered with filter paper. Put the index of the rack on the optimum porosity – Value. Then compress the sample between the two plugs by means of rack until the index moves to the top of the curve height of the sample. Insert the tube in the measuring circuit and set the apparatus working. When the level in the manometer is steady remount the rack and make it coincide with the lower portion of the meniscus of the liquid in the manometer. Read the value in the index on chart.

3.3 **Purity**

Dissolve 0.2 gm ZnO in hot 1 : 1 nitric acid.

Neutralize and add NH₄OH / NH₄Cl buffer. Titrate against 0.05 (%) M EDTA using Eriochrome black T indicator. (For details refer Analysis Text Book by A.I.Vogel).

**Calculation**

\[
\text{Wt. of sample} = W_1 \text{ gm} \\
\text{Volume of EDTA used} = V \text{ ml}
\]
Normality of EDTA = 0.05N

\[ V \times N \times 81.37 \]

Purity of Zinc oxide ( %) = \[ \frac{W1}{10} \]

4 DIOCTYL PHThALATE

4.1 Specific Gravity

Sp.gravity is found out using a specific gravity bottle. Take a clean dry specific gravity bottle and find its empty weight with stopper. Then fill it with the plasticizer and put the stopper taking care not to enter any air bubbles inside the bottle. Now wipe out the surface of the bottle to dry and find its weight. Then empty the bottle, clean and dry it and then fill the specific gravity bottle with distilled water. Put the stopper and wipe out the surface to dry and find out its weight. Calculate the sp.gravity of the sample.

\[ \text{Wt. of Sp. gravity bottle} = W1 \text{ gm} \]
\[ \text{Wt. of Sp. gravity bottle + sample} = W2 \text{ gm} \]
\[ \text{Wt. of Sp. gravity bottle + distilled water} = W3 \text{ gm} \]

\[ \text{Specific Gravity of sample} = \frac{(W2 - W1)}{(W3 - W1)} \]

4.2 Volatile Mater

Accurately weigh 20 ml of the sample in a previously dried and weighed petri dish. Keep in an oven at 105°C for 2 hrs. Take the dish out, cool in a desicator, and weigh. Find the loss in weight. Calculate the loss in weight and V.M. Run duplicate.

\[ \text{Weight of petri dish} = W1 \]
\[ \text{Weigh of dish + sample before heating} = W2 \]
\[ \text{Weigh of dish + sample after heating} = W3 \]

\[ \text{Volatile mater} (\%) = \frac{(W2 - W3)}{(W2 - W1)} \times 100 \]

4.3 Saponification value:

Weigh exactly 0.5 gm of the sample and transfer it into the 250 ml iodine flask. Then add exactly 25 ml of alcoholic potassium hydroxide of about 0.5N. Now fit it with air condenser and reflux on a water bath heated slow boiling until the reaction is complete (about 6 minutes). Allow to cool and wash the condenser using 2 ml distilled water. Then titrate with standard hydrochloric acid of 0.25N, in presence of phenolphthalein indicator. Similarly a blank also is to be carried out.
Calculation:

\[
\text{Sap.value (mg KOH/gm) } = \frac{(B - V)}{W} \times N \times 56.1
\]

Where

- \( V \) = Volume of \( \text{HC}1 \) used for titration in real test
- \( N \) = Normality of \( \text{HC}1 \)
- \( B \) = Volume of \( \text{HC}1 \) used for blank test
- \( W \) = Weight of the sample used for the test

4.4 Acid Value:

The sample dissolved in ethyl alcohol which is neutralized and titrated against standard sodium hydroxide using phenolphthalein as indicator.

Procedure:

In a clean dry conical flask transfer about 5 to 6 gm of sample exactly weighed. Add 50 ml of 1:1 toluene - methanol mixture, shake well for dissolution. Then titrate against standard 0.05 N alcoholic sodium hydroxide using phenolphthalein as indicator. Conduct a blank titration.

Calculation:

\[
\text{Acid Value (mg KOH/gm) } = \frac{\text{TV} - B \times N \times 56.1}{W}
\]

Where

- \( \text{TV} \) = Volume of KOH used for titration of sample
- \( N \) = Normality of Sodium hydroxide
- \( W \) = Weight of the sample taken for analysis
- \( B \) = Volume of KOH consumed for blank

4.5 MOISTURE CONTENT BY KARL FISHER TITRATION

Procedure:

Moisture in the plasticizer is determined by the Karl fisher method with Aquameter. Benzene is used for the media.

Apparatus:
Aquameter Beckman KF3 model or similar make

**Procedure:**
Check up the Aquameter for its perfect working condition. Then fill the Karl Fisher reagent bottle provided with the aquameter and fill the burette with Karl Fisher reagent. Take about 50 ml of pure anhydrous methanol in clean dry beaker provided with the aquameter and titrate with Karl Fisher reagent for anhydrous medium. Now, weigh exactly 10 micro liters of H₂O in a syringe. Of sodium titrate dihydrate in a weighing bottle and transfer it into the beaker containing anhydrous methanol. Now, operate the instrument and titrate against Karl Fisher reagent used for titration from this calculate how much mgs.H₂O is equivalent to one millilitre of Karl Fisher reagent. Weigh exactly about 5 grams of the sample in a clean dry weighing bottle and introduce into the beaker, stir well and titrate as before and note the volume of Karl Fisher used for titration. Now calculate the % of moisture as given below. For detailed operation procedure of the instrument refer to the instruction manual.

**Calculation:**
Let 1 ml of KF solution = mgs of H₂O and W be the wt. of sample taken and ‘V’ be the volume of Karl Fisher solution used for titration.

\[
X = \text{Strength of KFR} \\
X \times V \times 100 \\
\text{Moisture ( %) = } \frac{X \times V \times 100}{W \times 1000}
\]

5 COUMARONE INDENE RESIN

5.1 **Specific Gravity**
Specific gravity is found out using a specific gravity bottle with water.

Take a clean dry specific gravity bottle and find its weight with stopper accurately. Fill about 1/3rd of its volume with the sample. Put the stopper and find out the weight of the specific gravity bottle with the sample. Then fill the bottle 1/3 with water over the sample. Deaerate for half an hour to remove the air bubbles. Then fill with water, put the stopper, wipe out the surface of the specific gravity bottle with blotting paper and find the weight. Now empty the bottle, clean dry and fill it with distilled water. After putting the stopper with air bubbles wipe out the surface and note the weight with distilled water alone.

\[
W1 = \text{Wt. of specific gravity bottle} \\
W2 = \text{Wt. of specific gravity bottle + sample} \\
W3 = \text{Wt. of specific gravity bottle + sample + water} \\
W4 = \text{Wt. of specific gravity bottle + water} \\
\]

\[
\text{Specific Gravity} = \frac{(W2 - W1)}{(W4 - W1) - (W3 - W2)}
\]
5.2 **Softening point:**

5 gms of powdered resin is taken in a test tube. A small steel ball (bearing) is put over the resin and the test tube is heated slowly. The temperature at which the ball sinks in the resin is taken as the softening point.

6 **STEARIC ACID.**

6.1 **Specific Gravity:**

Specific gravity is found out using a Hubbard specific gravity bottle. Take a clean dry specific gravity bottle and find its weight. Fill about 1/3rd of its weight with the sample, put the stopper and find out the weight of the specific gravity bottle with the sample. Then fill the bottle with distilled water over the sample. Put the stopper, wipe out the surface of the specific gravity bottle with blotting paper and find the weight. Now empty the bottle, clean and dry and fill it with water alone and find its weight after putting stopper and wiping out the surface with blotting paper. Finally empty the bottle, clean dry and fill it with water. After putting stopper and wiping out the surface with blotting paper. Finally empty the bottle, cleaned dry and fill it with water. After putting stopper without air bubbles wipe out the surface and note the weight with distilled water alone.

\[
\text{Wt. of specific gravity bottle} = W_1 \text{ gm} \\
\text{Wt. of specific gravity bottle + sample} = W_2 \text{ gm} \\
\text{Wt. of specific gravity bottle + sample + water} = W_3 \text{ gm} \\
\text{Wt. of specific gravity bottle + water} = W_4 \text{ gm}
\]

\[
\text{Specific Gravity} = \frac{W_2 - W_1}{W_4 - W_1 - (W_3 - W_2)}
\]

6.2 **Melting point:**

Melting point is found out using a melting point apparatus.

6.3 **Purity:**

Weight exactly 0.5 gm of stearic acid in a clean dry conical flask. Add 50 ml carbon tetrachloride and shake well and if not clear add a little amount of Dioxan solvent. Add 2 or 3 drops of phenolphthalein indicator. It is then titrated against 0.05 N alcoholic potassium hydroxide.

\[
V \times N \times F \times 100 \\
Purity (\%) = \frac{V \times N \times F \times 100}{W \times 1000}
\]
V = Volume of alcoholic KOH
N = Normality of KOH
F = 284.49 (equivalent weight)
W = Wt. of sample taken

7 DIETHYLENE GLYCOL:

7.1 Boiling point

Boiling point of Diethylene glycol is found out using the boiling point apparatus.

The apparatus consists of a distilling flask attached by a cork to a Liebing condenser at the end of which an adapter is fitted. There is a receiver for collecting the distillate. A thermometer is fitted into the neck of the distilling flask by means of a well bored cork, the bulk of the thermometer should be in the centre of the neck of the flask and slightly below the level of the side tube. The flask may be heated on a wire quaze with asbestos center. The liquid is poured in the distilling flask and a few fragments of unglazed porous porcelain are added and the thermometer is placed in position. The flask is heated on a wire gauze. Heating may be rather rapid until boiling commences; the flame must then be decreased and adjusted so that the distillate is collected at the rate of one or two drops per second.

It will be found that the temperature will first rise rapidly until it is near the boiling point of the liquid then slowly and finally will remain practically constant. The distillation should be continued until only a small volume of liquid remaining in the flask, the temperature is noted at regular intervals. This constant temperature is the boiling point of the liquid.

7.2 Specific gravity

Take a clean dry specific gravity bottle of about 25 ml capacity and note the exact weight of it along with the stopper. Fill the bottle with distilled water and put the stopper carefully so that no air bubble is left inside. Wipe off the water on the outer surface of sp.gravity bottle and weigh it accurately. Empty the bottle and dry it in an air oven. Fill the bottle with diethylene glycol, put the stopper, clean the surface and weigh it again.

\[
\text{Sp.gravity} = \frac{(W3 - W1)}{(W2 - W1)}
\]

W1 = Wt. of Sp.gravity bottle
W2 = Wt. Of Sp.gravity bottle + water
W3 = Wt. Of Sp.gravity bottle + Diethylene glycol

8 BENZOTHIAZYL DISULPHIDE

8.1 Specific gravity

Specific gravity is found out using a specific gravity bottle with water.
Take a clean dry specific gravity bottle and find its weight with stopper accurately. Fill about 1/3\textsuperscript{rd} of its volume with the sample. Put the stopper and find out the weight of the specific gravity bottle with the sample. Deaerate for half an hour to remove the air bubbles. Then fill with water, put the stopper, wipe out the surface of the specific gravity bottle with blotting paper and find the weight. Now empty the bottle, clean dry and fill it with distilled water. After putting the stopper without air bubbles wipe out the surface and note the weight with distilled water alone.

\begin{align*}
\text{Wt.of specific gravity bottle} & \quad = \quad W1 \text{ gm} \\
\text{Wt.of specific gravity bottle + sample} & \quad = \quad W2 \text{ gm} \\
\text{Wt.of specific gravity bottle + sample + water} & \quad = \quad W3 \text{ gm} \\
\text{Wt.of specific gravity bottle + water} & \quad = \quad W4 \text{ gm} \\
\end{align*}

\[
\text{Specific Gravity} = \frac{(W2 - W1)}{(W4 - W1) - (W3 - W2)}
\]

9.1 **TETRAMETHYL THIURAM DISULPHIDE (TMTD)**

9.1.1 **Specific gravity**

Specific gravity is found out using specific gravity bottle with water. Find the weight of a clean dry empty specific gravity bottle. Fill about 1/3\textsuperscript{rd} of its weight with the sample. Put the stopper and find out the weight of the specific gravity bottle with the sample. Then fill the bottle with distilled water over the sample. Put the stopper, wipe out the surface of the specific gravity bottle with blotting paper and find the weight. Now empty the bottle, clean and fill it with water alone and find its weight after putting stopper and wiping out the surface with blotting paper. Finally empty the bottle clean, dry bubbles wipe out the surface and note the weight with distilled water alone.

\begin{align*}
\text{Wt.of specific gravity bottle} & \quad = \quad W1 \text{ gm} \\
\text{Wt.of specific gravity bottle + sample} & \quad = \quad W2 \text{ gm} \\
\text{Wt.of specific gravity bottle + sample + water} & \quad = \quad W3 \text{ gm} \\
\text{Wt.of specific gravity bottle + water} & \quad = \quad W4 \text{ gm} \\
\end{align*}

\[
\text{Specific Gravity} = \frac{(W2 - W1)}{(W4 - W1) - (W3 - W2)}
\]

9.2 **Total sulphur content:**

1 gm of the material is burnt in oxygen in a bomb. The sulphate formed is determined gravimetrically as barium sulphate.

Weigh exactly 1 gm of the material and burn it in oxygen in a bomb. The residue is dissolved in sodium hydroxide. Add HC\textsubscript{1} to the solution and boil. Then add about 20 ml of 10\% Barium chloride solution. Stir well and allow to settle for two hours. Now carefully filter through a filter paper (whatman No.42) for fine precipitate. Wash the precipitate till all the chloride ion is removed.
Dry the filter paper with the funnel in an oven at 100°C. Then take out and place the filter paper in a platinum crucible which is cleaned, dried and weighed previously. Heat the crucible till the filter paper is completely charred. Then after cooling add one or two drops of 50% sulphuric acid in the crucible and heat again. Then transfer the crucible into a muffle furnace and calcine for at least one hour at 800°C. Allow to cool in a desiccator and weigh.

\[
\text{Sulphur (\%) } = \frac{P1}{P} \times 13.7
\]

Where \( P = \text{Wt. of the test sample} \)
\( P1 = \text{Increase in wt. of the Platinum crucible} \)

10 **NBR (Medium high acrylonitrile)**

10.1 **Specific gravity**

Specific gravity is found out using Hubbard specific gravity bottle with water. Take a clean dry specific gravity bottle and find its weight. Fill about 1/3rd of its weight with the sample, put the stopper and find out the weight of the specific gravity bottle with the sample. Then fill the bottle with distilled water over the sample, put the stopper, wipe the surface of the specific gravity bottle with blotting paper and find the weight. Now, empty the bottle, clean and fill it with water alone and find its weight after stoppering and wiping out the surface with blotting paper.

**Calculation:**

\[
\text{Wt. of specific gravity bottle} = W1 \text{ gm}
\]
\[
\text{Wt. of specific gravity bottle + sample} = W2 \text{ gm}
\]
\[
\text{Wt. of specific gravity bottle + sample + water} = W3 \text{ gm}
\]
\[
\text{Wt. of specific gravity bottle + water} = W4 \text{ gm}
\]

\[
\text{Specific Gravity} = \frac{(W2 - W1)}{(W4 - W1) - (W3 - W2)}
\]

10.2 **Acrylonitrile content**

One gram of finely shredded NBR is digested with con.H\(_2\)SO\(_4\). The ammonium salt formed is estimated as NH\(_3\) using Kjeldhal's method. Calculate the percentage of Nitrogen as acrylonitrile percentage.

Take .3 gm of the sample in a 1 litre Kjeldal flask. Add 20 cc of con. H\(_2\)SO\(_4\) 10 gms of potassium sulphate and 0.5 gm of copper sulphate. Heat the flask strongly in a fume cupboard using a burner for several hours. When the solution becomes clear, stop heating, cool and dilute it with 100 ml distilled water. Connect the flask with Kjeldhal's apparatus used for estimating nitrogen.
Add 100 ml of 30 – 10% NaOH through the funnel and start heating. Absorb the ammonia evolved in 50 ml 0.1 \( \text{H}_2\text{SO}_4 \) solution. Continue heating for one hour and titrate the excess acid against 0.1N NaOH using methyl red indicator. Conduct blank experiment. From the volume of NaOH calculate:

\[
\text{Acrylonitrile content} \% = \frac{\text{B} - \text{TV} \times N \times 53 \times 100}{\text{W} \times 1000}
\]

Where

- \( \text{N} \) = Normality of Alkali
- \( \text{TV} \) = Volume of Alkali consumed for the sample
- \( \text{B} \) = Volume of Alkali consumed for the blank
- \( \text{W} \) = Weight of sample

_Schematics of the Insulation Drawings of different Hardwares are depicted following_

Hardware No.-01 (S200 HES)
Hardware No.-02(S200 MS)
Hardware No.-03 (S200 NES)
Hardware No.-04 (PSOM XL HES)
Hardware No.-06 (PSOM XL NES)
Hardware No.-07    (S200 BFL)