Document No.: HEMRL/TRM/PROP/RM/4[REVISED]

SPECIFICATION FOR

HYDROXY TERMINATED POLYBUTADIENE

(PROVISIONAL)

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0. FOREWORD

0.1 This specification has been prepared by High Energy Materials Research Laboratory, Sutarwadi, Pune - 411021

0.2 This specification will be approved by the Ministry of Defence after appropriate sealing action by Controllerate of Quality Assurance (Military Explosives) and will be mandatory for use by Defence Services.

0.3 Before sealing action, any queries regarding this specification may be referred to High Energy Materials Research Laboratory, Sutarwadi, Pune - 411021

1. SCOPE

1.1 This specification is intended to govern, supply and Quality Assurance of Hydroxy Terminated Polybutadiene (HTPB) with antioxidant (Hydroquinone).

1.2 INTENDED USE

This material is intended for use as an ingredient in the booster and sustainer propellants of "TRISHUL" Missile/Pinaka/RZ-61/Pechora/Akash Booster.

2 RELATED SPECIFICATIONS AND DOCUMENTS

2.1 The following related specification has been referred to in the preparation of this specification.

MIL-H-85497 (AS) HYDROXY TERMINATED POLYBUTADIENE
6th Oct 1981

Any queries regarding this MIL standard specification may be referred to High Energy Materials Research Laboratory, Sutarwadi, Pune - 411021

3 MATERIAL / FINISH

The material shall be in the form of clear viscous liquid.

4 MANUFACTURE

4.1 Hydroxy Terminated Polybutadiene shall be manufactured by a process which will produce the product conforming to this specification.

4.2 Nothing in this specification shall relieve the manufacturer of his responsibility for the safety of his operations.

5. TENDER SAMPLE

5.1 The contractor / supplier shall submit a tender sample of 1 Kg free of each individual (primary sample) batch and 1 Kg from the blended lot (composite sample). Acceptance of the tender will denote that the tender sample is accepted as a standard of supply in accordance with the terms of this specification.

6 QUALITY ASSURANCE

6.1 INSPECTION

6.1.1 Hydroxy Terminated Polybutadiene and the containers in which it is packed shall be subject to inspection by and to the final approval of the Quality Assurance Officer / Quality Assurance Authority.

6.1.2 Samples of the material may be taken from any portion of the batch / lot / consignment.

6.1.3 If on examination any sample be found not to conform to this specification the whole batch / lot / consignment may be rejected.

6.2 SAMPLING

Chemical/Physical properties tests shall be run on each primary sample (individual batch) and composite sample (blended lot). Glass containers shall be used for all liquid samples. Each sample shall be labelled with date, lot number, and manufacturer's container identification number.

6.3 TEST REQUIREMENTS

6.3.1 Samples taken from any portion of the batch / lot/ consignment of the material shall conform to clause 3 and in addition shall conform to the following test requirements. (Applicable to all primary and composite samples).

Tests:

Sl No.	Characteristics	Passing Standard	Reference to Test Method
1	Hydroxyl Value, mg of KOH/g of sample	40 – 45	Appendix - RM/4[R]/I BM 3020/87 (Ref. ASTM D 2849)
2	Acid Number, Max. mg of KOH/g of sample, Max.	1.0	Appendix - RM/4[R]/II BM 3022/87 (Ref. ASTM D 2849)
3	Volatile matter, percent by mass, Max	0.5	Appendix - RM/4[R]/III BM 3009/86 (Ref. ASTM D 3030)
4	Viscosity at 30^0c, Poise Viscosity at 60^0c, Poise	40 –65 9 – 16	Appendix - RM/4[R]/IV BM 3021/87 (Ref. ASTM D 2393)
5	Specific gravity, at 30^0 C	0.9 – 0.92	Appendix - RM[R]/4/V BM 3010/86 (Ref. ASTM D 792)
6	Moisture content, percent by mass, Max.	0.1	Appendix - RM/4[R]/VI BM 3023/87 (Ref. ASTM E 203)
7	Molecular weight, (Mn), [VPO}	2300 to 2900	Appendix - RM/4[R]/VII BM 3026/88 (Ref. Knauer VPO Manual)
8	Polydispersity (GPC)	1.5 – 2.5	Appendix - RM/4[R]/VIII
9	Intrinsic Viscosity, dl/g	0.11 – 0.17	Appendix - RM/4[R]/IX

6.4 STABILITY

When packed in accordance with clause 9 and stored at temperature less than 38^0C the HTPB shall meet the requirements of this specification for a minimum period of 12 months after acceptance. The shelf life may be extended by 6 months at a time after reacceptance testing for conformance to requirements of hydroxyl value, moisture, viscosity and acid number.

6.5 Toxic Products: - Safety regulations and guidelines applicable to the use of HTPB should be complied with to preclude personal injury and damage to equipment and facilities.

7 SUPPLIERS INSPECTION OF STORES / CONSIGNMENT

7.1 Before tendering the store for inspection the supplier shall carry out a through inspection of each delivery to satisfy himself that the store fully conforms to this specification and shall render certificate to that effect to the Quality Assurance Officer / Quality Assurance Authority.

8 WARRANTY

8.1 The stores supplied against the contract shall deem to have been warranted against defective material and performance by the contractor / manufacturer for a period of 12 months from the date of receipt of the stores at the consignee's end and if during this period any of the stores supplied is found defective the same shall be replaced by the contractor / manufacturer free of charge at the consignee's premises.

9 PACKAGING

9.1 Hydroxy Terminated Polybutadiene shall be packed in suitable galvanized steel drums.

9.2 When the material is required to be transported by rail the packing shall conform to the provisions of Indian Railways Conference Association, Red Tariff No.18.

9.3 The inclusion of any foreign matter or impurities in any of the packages shall render the whole consignment liable to rejection.

10 MARKING

10.1 All packages containing the material shall be durably and legibly marked with the following details (as applicable):

i) Nomenclature and specification number of the material.
ii) Name and address of the consignee.
iii) S.O. Number and date
iv) Consignment number

v) Lot / Batch number and date of manufacture

vi) Gross and net mass

vii) Consecutive number of package and total number of packages in the consignment

viii) Date of Supply

ix) Contractor's initials or recognized trade mark

x) Storage temperature limit.

10.2 In addition to the above the Quality Assurance Officer / Quality Assurance Authority may suggest some more markings / identifications considered suitable at the time of inspection.

10.3 The paint used for marking shall conform to IS :138 –1981 and to the satisfaction of the Quality Assurance Officer / Quality Assurance Authority.

11 DEFENCE STORES CATALOGUE NUMBER

11.1 Defence Stores Catalogue Number allotted to this store is Nil

12. SUGGESTIONS FOR IMPROVEMENT

12.1 Any suggestion for improvement in this particular document may be forwarded to High Energy Materials Research Laboratory, Sutarwadi, Pune - 411021

13. APPENDICES

APPENDIX - RM/4[R]/I

HYDROXYL VALUE

1. APPARATUS

a) Iodine flasks, 250 ml.
b) Water condensers
c) Hot plate
d) Burette, 50 ml
e) Pipettes, 20 ml, 10 ml

2. CHEMICALS

a) Acetic Anhydride in Pyridine
Mix 25 ml of acetic anhydride (Analar) in 200 ml pyridine (Analar). Store in an amber colour bottle. Prepare fresh every day. Discard the reagent if the colour is darker than pale yellow.

b) Sodium Hydroxide – Standard 0.6 N
Standardize accurately against dried potassium hydrogen phthalate. Standardize regularly. Prevent exposure to CO_2 in the atmosphere.

3. PROCEDURE

Take three dry iodine flasks. Weigh into each flask. 5 to 8 g of HTPB to the nearest 0.1 mg. Use a glass tube and prevent sample from touching the ground joint part of the flask. Add 20 ml of acetylating reagent with a 20 ml volumetric pipette. Add a boiling chip or glass bead. Attach a water condenser to each iodine flask and place on hot plate. Use toluene to moisten the ground joint. Start the water circulation. Reflux the sample for one hour after reflux starts. Add 10 ml of distilled water by graduated pipette. Continue refluxing for 15 minutes. Cool the flasks, rinse the condenser completely with two 20 ml portions of 20 ml distilled water using graduated pipette. Disconnect the condensers, rinsing the ground joint with 20 ml distilled water at the same time collecting the water in the flask.

Add 1 ml phenolphthalein indicator using a 1 ml graduated pipette. Titrate against standard 0.6 N NaOH shaking well each time. Towards the end point, add 100 ml distilled water, mix well and continue titration.

After each addition shake well, allow to settle and observe the colour of the aqueous layer. The end point is reached when the colour just turns pinkish orange from the original pale yellowish colour. Do a blank in duplicate at the same time. Determine the acid number of the sample as mg KOH/g of sample (Appendix – VII).

4. CALCULATIONS

Hydroxyl number,

$$\text{mg of KOH/ g of sample} = \frac{(V_2 - V_1) \times N \times 56.1}{m} + A$$

Where,

V_1 = Net Titre for sample in ml

V_2 = Net Titre for blank in ml

N = Normality of NaOH used

m = Mass of the sample, g

A = Acid number of sample in mg of KOH/g

NOTE: Pyridine is corrosive and should be handled carefully. Prevent with skin. Acetic anhydride is an irritant and should also be used carefully. Avoid inhalation of these chemicals and prepare the reagent in the fume cupboard.

APPENDIX –RM/4[R]/II

ACID NUMBER

1. **APPARATUS**

a) Burette, 10 ml graduated in 0.05 ml divisions

b) Erlenmeyer flasks

c) Pipettes – graduated 10 ml and 1 ml.

2. **REAGENTS**

a) Titration solvent: Benzene / Isopropyl alcohol (IPA) mixture (2:1 ratio by volume)

b) Standard 0.1 N NaOH (aqueous)

c) Phenolphthalein Indicator 1% m/v in IPA.

3. **PROCEDURE**

Take 100 ml titration solvent into a 250 ml Erlenmeyer flask with a 100 ml measuring cylinder. Add 6 to 8 g of the sample to the nearest 0.01 g. Mix it by swirling it. Add Phenolphthalein indicator (0.5 ml with pipette). Mix by swirling. Titrate the sample with 0.1 NaOH from a 10 ml burette to the first faint pink colour. Note the titration reading. Carry out blank repeating same procedure. Note the time reading. Calculate acid number.

4. **CALCULATIONS**

$$\text{Acid Number Mg of KOH/g of Sample} = \frac{(V_1 - V_2) \times N \times 56.1}{m}$$

Where

V_1 = Net titre for the sample, ml

V_2 = Net titre for the blank, ml

N = Normality of NaOH used

M = mass of sample, g

NOTE : Benzene / IPA mixture is inflammable. Keep away from lot surfaces and avoid contact with hands. In case of contact with skin, wash with plenty of water.

APPENDIX – RM/4[R]/III

VOLATILE MATTER

1. APPARATUS

a) Oven maintained at 105 ± 1^{0}C

b) Petri dish, dia 55 mm, depth 15 mm

c) Mettler balance with accuracy of 0.1 mg.

2. PROCEDURE

Dry the clean empty petri dish in the oven at 105 ± 1^{0}C for 30 min. Cool in a desiccator and weigh (to the nearest 0.1 mg). Transfer about 5 g of HTPB and weigh accurately to the nearest of 0.1 mg. Keep the dish with sample in the oven at 105 ± 1^{0}C for 2 hours. Remove the dish and cool in a desiccator. Weigh the dish with the sample.

3. CALCULATION

Volatile matter, % by mass $= \dfrac{m_2 - m_3}{m_2 - m_1}$

Where,

m_1 = mass of the empty petri dish, g

m_2 = mass of the petri dish and sample, g

m_3 = mass of the petri dish and sample after drying, g

Report the results of two determinations and their average.

APPENDIX – RM/4[R]/IV

VISCOSITY

1. **APPARATUS**

a) Brookfield Viscometer – Model RVT with guard legs

b) Spindle No.4

c) Water jacketed sample container with Thermostatic bath

d) ASTM 120 Thermometer

2. **PROCEDURE**

Sample into the jacketed sample container by pouring approximately 400 ml, carefully into the container along the sides. Avoid air bubbles in the sample. Adjust temperature to 30^0C by circulating water from the thermostat bath through the jacket of the container. Stir the contents of the container using the thermometer and note the temperature at the centre. Stirring should be done carefully avoiding formation of air bubbles.

Read the instruction manual prior to use. When the temperature is 30^0C ± 0.1^0C, introduce the spindle No.4 into the sample and stir sample with the spindle. Check the temperature (30^0C ± 0.1^0C). Adjust the viscometer (with guard leg) so that the spindle is at the centre and immersed to the mark on the stem. Adjust the level of the viscometer using the 3 levelling screws and the spirit level. Switch on the viscometer, and adjust it for zero setting. Set the speed selector by using the speed selector knob so that maximum dial reading is possible (but less than 90) using 20 RPM. Note the dial reading when the pointer position is constant.

Repeat the experiment at 60^0C ± 0.1^0C. Adjust the thermostat bath temperature of the sample to 60^0C ± 0.1^0C. Stir the sample with the thermometer and note the temperature at the centre. Repeat the procedure as given for 30^0C. (However R.P.M. should be 50).

Calculate the viscosity at 30^0C and 60^0C in centipoises (cps).

APPENDIX –RM/4[R]/V

SPECIFIC GRAVITY

1. **APPARATUS**

   a) Analytical balance with 0.1 mg accuracy

   b) Beaker, 250 ml

   c) Metallic sinker (approx. 50 g) with wire for suspending from the pan of the balance.

2. **PROCEDURE**

Keep a part of the sample (approx 200 ml) in the 250 ml beaker in the constant temperature room at 23 ± 1^{0}C till it attains constant temperature. Suspend the sinker by means of the wire from hook at the top of the balance and weigh it to the nearest of 0.1 mg.

Remove the sinker, place the pan straddle across the pan. Place a clean dry 250 ml beaker filled with 200 ml distilled water on top of it (at 23 ± 1^{0}C) avoiding any air bubbles and suspend the sinker from the hook taking care to see that the sinker is completely immersed and it is not touching the sides or bottom of the beaker. Weigh the sinker in water. Carefully remove the beaker and sinker. Remove the sinker, wash with acetone and dry. Find the weight of the sinker in HTPB resin as above but using HTPB at 23 ± 1^{0}C instead of distilled water.

3. **CALCULATIONS**

Specific gravity at, 23/23 deg C $= \dfrac{m_1 - m_3}{m_1 - m_2}$

Where,

m_1 = mass of sinker + wire in air, g

m_2 = mass of sinker + wire in water, g

m_3 = mass of sinker + wire in HTPB, g

Report the results of two determination.

APPENDIX – RM/4[R]/VI

MOISTURE CONTENT

1. **APPARATUS**

   a) Karl Fischer Apparatus

2. **REAGENTS**

   a) Karl Fischer Reagents F=6

   b) Benzene / Isopropyl alcohol (IPA) mixture – 1:1 by volume (moisture free)

   c) Pyridine (moisture free)

3. **PROCEDURE**

   Introduce 20 ml of pyridine into the titration vessel using a 20 ml pipette. Add 20 ml mixture of Benzene/IPA using a pipette. Start the stirrer and titrate with Karl Fischer Reagent till just neutralized. Add 10 g of sample and allow it to dissolve. Titrate the sample with Karl Fischer reagent. Note the titrate value. Calculate the moisture content.

4. **CALCULATIONS**

$$\text{Moisture content, Percent by mass} = \frac{V \times F}{10 \times m}$$

Where,

V = Titre volume for sample, ml

F = Karl Fischer, reagent factor – mgH_2O/ml

m = Mass of the sample, g

NOTE : Handle Pyridine, Benzene / IPA and Karl Fischer reagent mixture carefully. Avoid inhalation and spillage. Dispose these chemicals into the carbuoy kept for this purpose.

APPENDIX – RM/4[R]/VII

DETERMINATION OF THE NUMBER AVERAGE MOLECULAR WEIGHT (M_n) OF HTPB RESIN USING A KNAUER VAPOUR PRESSURE OSMOMETER

SCOPE

This method describes a procedure for determining the Number Average Molecular Weight (M_n) of HTPB using a Knauer Vapour Pressure Osmometer.

SUMMARY

A Kaneuer Vapour Pressure Osmometer (VPO) used for this determination, has two matched thermistors suspended in a temperature controlled cell containing Toluene. The thermistors form part of a Wheatstone bridge. The bridge is balanced after introducing identical drops of Toluene on both the reference and sample thermistors. The solvent drop on the sample thermistors is then replaced by a solution drop. Since the Vapour Pressure of the solvent in the solution drop is lowered due to the presence of the solute, solvent vapour condenses on the solution drop resulting in a change in temperature of the thermistors and hence its resistance. This gives an electrical output signal. The instrument is first calibrated using a series of solutions of known concentrations of a standard substance of known molecular weight in order to determine the instrument constant. A series of solutions of HTPB are then used. The molecular weight is determined by the procedure given below:

SAFETY

Follow good laboratory practices.

Dispose hydrocarbons in a carboy meant for this purpose.

APPARATUS

Knauer VPO with Universal thermistors and six syringes and a recorder with 100 mV FSD.

CHEMICALS

Calibration Standard: Squalane (Molecular Weight = 422.8) Chromatography Grade Toluene Analar.

PROCEDURE

A. SETTING UP THE KNAUER VPO

Note.1 : Read the Instruction Manual before using the VPO

2 : Allow the instrument to stabilize for at least two hours before using the VPO.

a. Clean and dry the inner chamber and wick using fresh Toluene.

b. Insert the wick into the chamber and fill the chamber with 20 ml fresh Toluene. Place the inner chamber in the instrument and fit the cell lid.

c. Set the operating temperature to 65^0C. Allow the instrument to stabilize for at least 2 hours. Stability is obtained when the instrument read-out is stable and the recorder baseline is stable.

B. PREPARATION OF STANDARD AND SAMPLE SOLUTIONS

1.0 Preparation standard solutions of 0.6, 0.8, 1.0, 1.2 and 1.0% m/m

1.1 In a 100 ml stoppered conical flask,. Weigh 0.8 gm Squalane to the nearest 0.1 mg. Add Toluene so that the weight of the solution is about 40.0 g. Weigh the solution to the nearest 0.1 mg. Mix well. Calculate the exact concentration (% m/m) to 4 decimals.

1.2 Prepare dilute solutions of 0.6, 0.8, 1.0, 1.2 and 1.4% m/m by dilution of a known amount of the solution in 1.1 with a known amount of Toluene in 10 ml volumetric flask. Weigh both the amounts of the concentrated solution and the diluted solution to the nearest 0.1 mg. Mix well. Calculate the concentrations of the different solutions to 4 decimals.

2.0 Prepare sample solutions of 2.0, 3.0, 4.0 and 5.0 % m/m

2.1 Into a 100 ml stoppered conical flask weigh 1.8g HTPB to the nearest 0.1 mg. Add Toluene so that the weight of the solution is about 32.0 g and weigh to the nearest 0.1 mg. Mix well. Calculate the exact concentration (% m/m) to 4 decimals.

2.2 Dilute appropriate quantities of the solution in 2.1 with known amount of Toluene in a 10 ml volumetric flask. Weigh both the concentrated and dilute solutions to the nearest 0.1 mg. Calculate the concentrations (% m/m) of the different diluted sample solutions to 4 decimals.

C. CALIBRATION

1. Adjust the Bridge Supply Voltage to 100% by turning the % Dial knob clockwise to extreme right. Press the Bridge Voltage button; the reading should be 100.0

2. Fill two clean and dry syringes with 1.0 ml Toluene, attach the needle and springs and insert them in the unnumbered apertures. Fill up the other four syringes with the diluted standard solutions and insert them into apertures numbered 1 to 4 so that the lowest concentrated solution is in aperture 1 and the solutions with increasing concentration levels are inserted in numerical sequence from the lowest to the highest.

3 Set the Measuring Bridge to zero by rinsing both the thermistors with pure solvent and take a drop of solvent on each thermistors. After 3-4 minutes or when the baseline is stable. Adjust the zero Balance (Coarse and Fine Zero Balance) to get a reading of 0.0 on the digital read-out meter keeping the Range switch at 2. Repeat this three times applying fresh drops each time.

4. To obtain reproducible result, it is necessary to rinse the thermistors before forming equal size drops. Rinsing volume and time interval between dripping and reading should remain constant.

Before injecting, push down the syringe with one hand. With the fingers of the other hand lightly turn the plunger while slowly pushing it down. Injection should cease when a drop first falls from the spiral end of the thermistors. The solution droplet remaining on the thermistors is then always uniform.

5. Set the Range switch to 8. Inject standard solutions starting always with the most dilute solution and using the dripping technique solution and using the dripping technique described in the previous paragraphs.

Use the following steps:

a) Rinse the left thermistors slowly with 10 drops of standard solution.

b) Take a fresh solvent drop on the right thermistors.

c) Take a solution on the left thermistors. Immediately after taking a drop on the left thermistors, press the right and start button of the timer. After the expiry of 4 minutes, the reading will be held in the display until the start button is pressed again. Note the reading.

Repeat steps (a) to (c) till a constant reading (+/- 0.5) is obtained thrice. In this case use only 2 drops in step (a). Note the reading.

d) Repeat step 5 with each of the solutions. Note the reading each time.

D. **ANALYSIS**

Set the range knob to 8.

Replace the standard solutions in the syringes with sample solutions (after cleaning and drying them).

Repeat steps 1 – 5

DETERMINING THE INSTRUMENT CONSTANT

Tabulate the readings for the standard solutions as follows:

No.	Solution % m/m	Conc (C)	Readings Reading (V)	Mean	V/C

Plot a graph of concentration (C) % m/m, on the x-axis, against V/C on the Y-axis. Draw the best, fit straight line and extrapolate the line to the Y-axis. Note the Intercept on the Y-axis = Ia.

Calculate the Instrument Constant K = Ta X 422.8

CALCULATION OF THE SAMPLE MOLEDULAR WEIGHT (M_n)

Tabulate the readings for sample solutions as in paragraph (E) and plot the graph.

Let the Intercept = Ib

Calculate the sample Molecular Weight

Molecular weight (M_n) = K/Ib

Report the Molecular Weight (M_n) to the nearest 100

The intercepts Ia or Ib are also calculated using a Scientific Calculator; ignoring those points which are away from the straight line. The slope and co-relation coefficient are also checked.

Always use a recorder to check that the readings obtained are stable values. If the recorder trace is still drifting after 4 minutes, the readings cannot be used. In this case clean the thermistors and start a fresh.

APPENDIX – RM/4[R]/VIII

POLYDISPERSITY (GPC)

Instrument: Gel Permeation Chromatography (GPC) System with UV & IR detector

GEL PERMEATION CHROMATOGRAPHY (GPC) is a liquid column chromatographic technique used to determine weight average (M_w) and number average (M_n) molecular weights of polymeric material (HTPB), where polymer (HTPB) molecules are separated according to their hydrodynamic volume or size. Diffusion of HTPB molecules takes place between mobile phase and the pores of the micro porous HTPB gels at the column. Mn & Mw distribution is determined from the measured retention volume by means of a calibration curve and polydispersity can be calculated by using the following equation:-

$$\text{Polydispersity} = \frac{M_w}{M_n}$$

APPENDIX – RM/4[R]/IX
INTRINSIC VISCOSITY

General

This method covers the determination of viscosity of dilute solution of HTPB and direction for calculating the intrinsic viscosity. Intrinsic viscosity is determinate by measuring the efflux time of dilute solutions of resins in toluene using Canon-Ubbelhode viscometer.

Apparatus:

a) Viscometer: A Canon –Ubbelhode viscometer giving an efflux time of not less than 35 s for solvent at 30^0C

b) Constant temperature bath: Capable of maintaining the temperature ± 0.10C at 30^0C

c) A standard thermometer having a range of 0-100^0C and an accuracy of ±0.1^0C

d) Timer: A timer, which can measure the correct time up to 0.1s

e) Volumetric flasks: 50 ml & 100 ml

Reagent

a) Toluene: AR Grade

Procedure:

Preparation of Stock solution: 2% w/v

Weigh accurately 2 g of sample into 100 ml volumetric flask and dissolve it in 50 ml of toluene. The solution must be absolutely free from undissolved matter or gel. Keep the flask (30^0C) and dilute up to the mark using toluene.

Preparation of different concentration:

Prepare 50 ml each of solution concentration of 0.25%, 50%, 0.75%,1.00%,1.25% and 1.50% by properly diluting the stock solution with toluene as described above by following equation.

$$\text{Volume of stock solution required} = \frac{\text{Required \% of conc. X required volume}}{\text{Weight of sample taken for stock solution}}$$

Procedure

Set the viscometer in the temperature bath maintained at the test temperature (30°C). Transfer the solvent (toluene) in the viscometer and keep in to the bath to attain the temperature equilibrium. Bring the solvent level in the viscometer above the graduation mark by applying suction at the limb opposite to the capillary. Allow the liquid to drain down through the capillary, start the timer exactly when meniscus passes the upper graduation mark and stop it when the meniscus passes the lower mark. Determine the efflux time t_0 for the solvent at least three times. The reading should agree within 0.1 s or 0.1%, for their mean, whichever greater.

Rinse and charge the viscometer with solution of lowest concentration (0.25%), repeat the experiment as above and find efflux time(t_1), find the efflux time (t_2 to t_6) for the remaining conc. as above in the ascending order of conc.

Calculation

Calculate the reduced viscosity of HTPB by following equation

$$\text{Reduced viscosity } (\eta_{red}) = \frac{t - t_0}{t_0 - C}$$

Where,

t = efflux time for sample (t_1, t_2, -------)

t_0 = efflux time for solvent

C = Concentration, %

Intrinsic viscosity (η)

Plot reduced viscosity Vs conc. and extrapolates to zero conc. and report the intrinsic viscosity in dl/g

This document contains 25 pages & issued to [illegible] as requested vide L.No. LY/23)PROJ/BF, dated 19th Feb 2013

20/02/2013