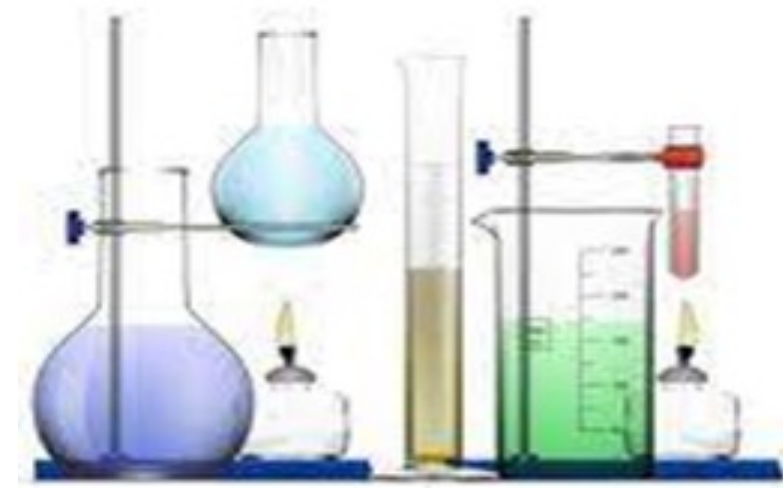




गुणवत्ता नियंत्रण प्रयोगशाला/ Quality Control Laboratory
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**ESTABLISHMENT OF METHOD: IS 10512
DETERMINATION OF WAX CONTENT
IN BITUMEN**



Established Wax Content in Bitumen VG40-IS 10512, first time in India

Compliance of MORTH Circular issued on 23.08.2023

Background: As per MORTH circular Clause No. 2.2.1 Dated 23.08.2023, Absolute Viscosity of VG40 should be min 3600 Poise. In addition to above, Bitumen should be tested for wax content as per IS: 10512 and wax content shall not be more than 4.5%.

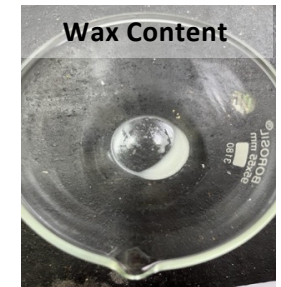
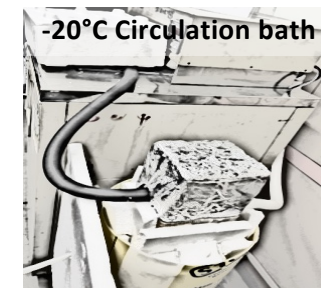
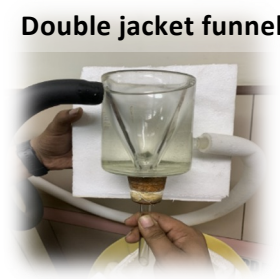
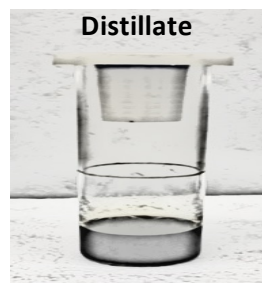
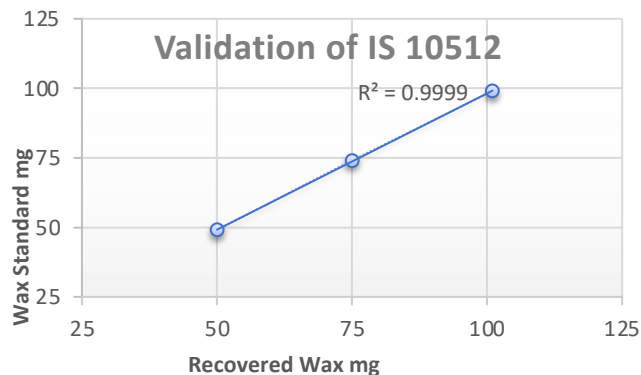
Test Facility Status: As the test facility is neither available nor the apparatus is readily available in the Indian market as per IS: 10512.

Methodology of Experiment: The major bottleneck was crystallization of bitumen distillate at -20°C as per IS 10512. To achieve the same, special double jacket glass funnel was cooled using -20°C solvent using circulation bath (-20°C) for crystallization of wax in bitumen extract. More than 10 samples tested. In IS 10512 cooling is achieved by manually adding CO₂ & Acetone mixture.

Method Validation: Method was validated by adding known mass of paraffin wax and again crystalized as per IS 10512. Obtained results are repeatable and recovery was 99%. Regression coefficient (correlation between standard value Vs obtained result) i.e. r^2 is 0.9999 which indicates excellent value of standardization.

Self Resilient: Thus HRQC innovatively established wax determination method in Bitumen as per IS10512 using 100% make in India facility and become first to achieve this rare test method. Also set an example of self resilient and Make in India initiatives at Haldia.

Results: Bitumen VG40 wax content is $<1.5\%$. This way HRQC provided solution to existing challenge for wax content in India for the first time.



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National Test Facility Status: As the test facility is neither available nor the apparatus is readily available in the Indian market as per IS: 10512.

Oversea Test Status: At present Intertek Singapore has test facility as per IS10512. IOC is sending sample to Singapore for wax content.

Challenge and Solution: Haldia Refinery took initiatives for this challenging task and explored for establishing this unique method with existing infrastructure and facilities. After a numbers of trials, finally got success to determine wax content as per IS 10512.

IS10512

IS 10512 : 2003

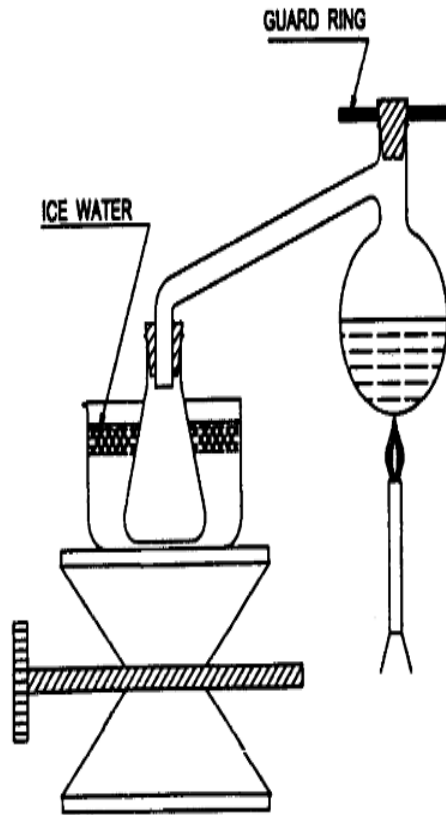


FIG. 3 SCHEMATIC DIAGRAM OF DISTILLATION ARRANGEMENT

IS 10512: COMPLIANCE AT HRQC



Distillation Flask as per Method

Fig1. Distillation assembly as per IS 10512

To obtain desired flame height (150 mm) use LPG with controlled air mixture.

IS10512

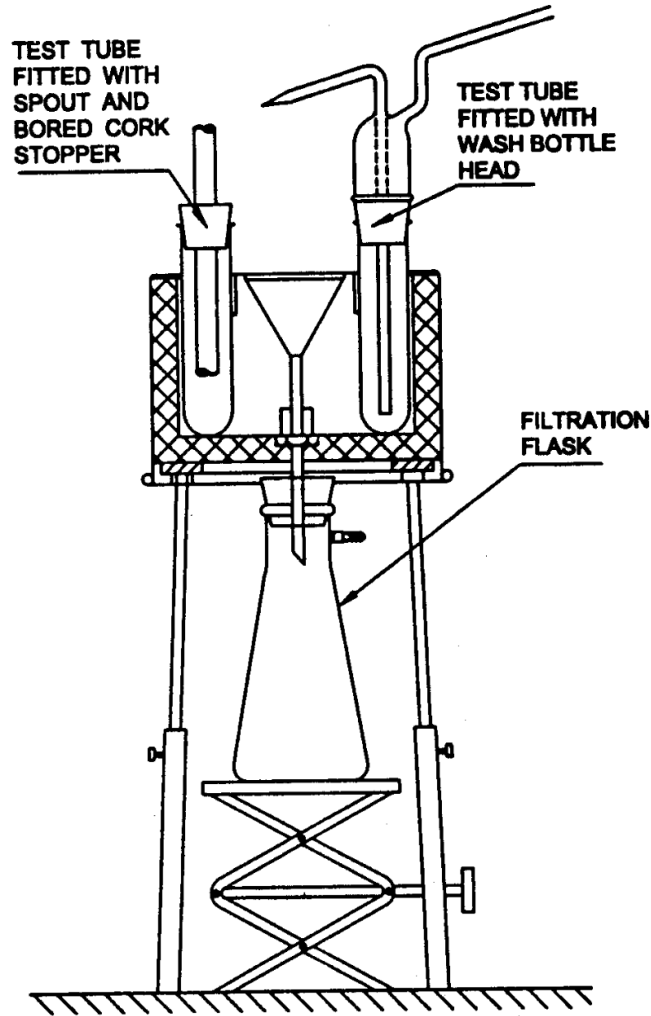


FIG. 4 SCHEMATIC DIAGRAM OF FILTRATION ARRANGEMENT

IS 10512 COMPLIANCE AT HRQC

Filtration of distillate with solvent (diethyl ether + ethanol mixture) at -20°C as per Method

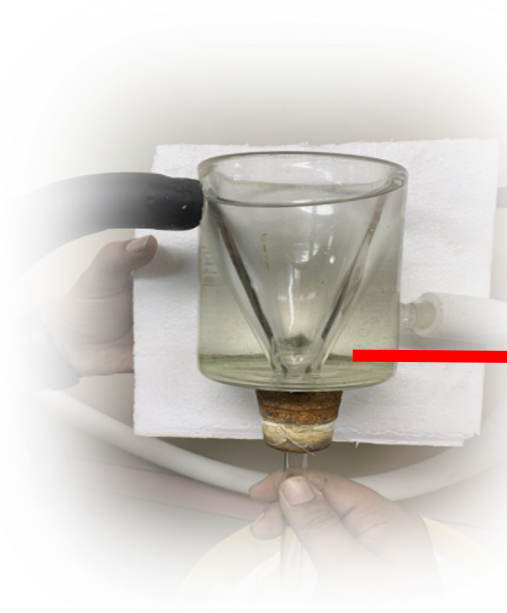


Fig 2. Basic Concept



Wax content

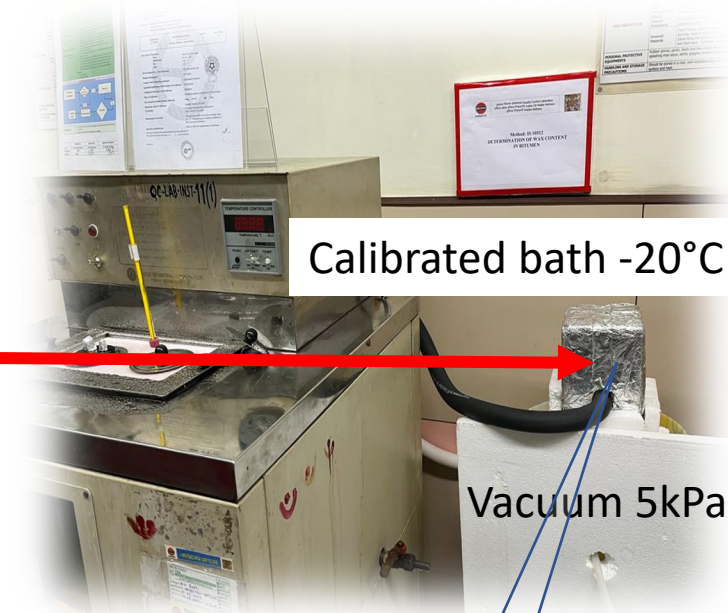


Fig 3. Insulated assembly at -20°C

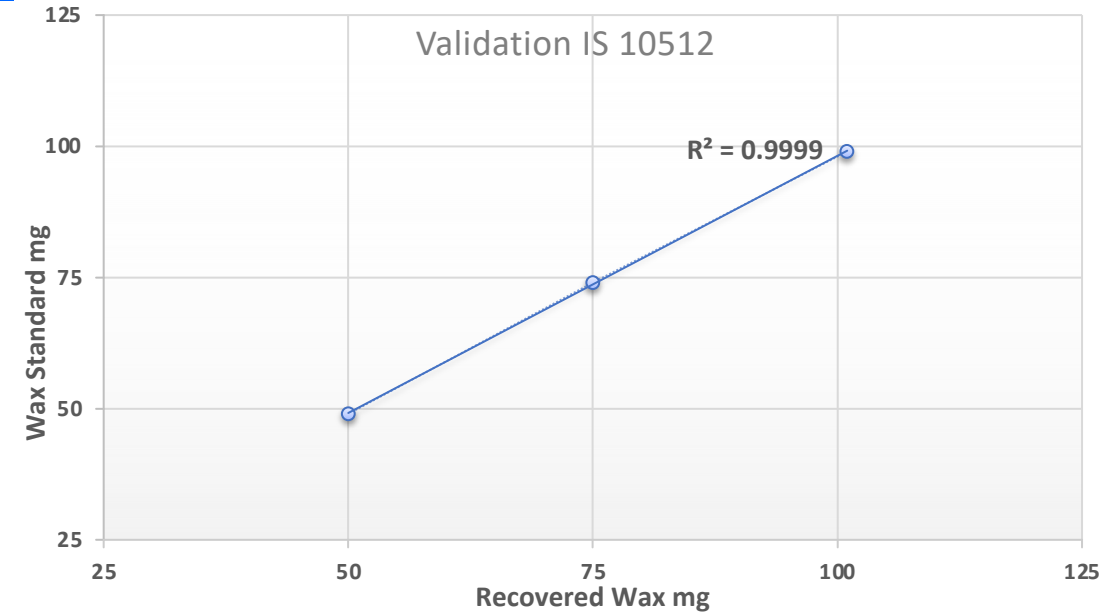
Filtration Assembly

-20°C maintained by CO2 and Acetone

Innovative Idea: -20°C maintained by calibrated circulation bath passed through double jacket glass funnel fitted on filtration flask.

Method Validation/Standardization

Validation of Method IS:10512	
Known Value	Observed Result
Blank sample	0 g
Blank +4g wax	4.0 g
Blank+ 4g wax	4.0 g
50mg	49mg
75mg	74mg
101mg	99mg

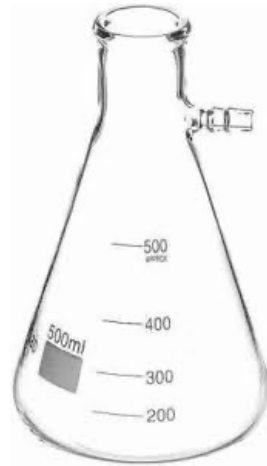


Sampling Date	Sample ID	Wax Content (%)
02-05-2023	TK 967	0.8
19-05-2023	TK 957	0.3
30.05.2023	TK 956	0.5
03-06-2023	TK 955	1.1
08-06-2023	TK 956	1.1
14-06-2023	TK 966	1.0
18-06-2023	TK 958	1.0
21-06-2023	TK 967	1.1
04-09-2023	TK 956	0.3
04-09-2023	TK 955	0.3
27.9.23	TK 967	0.5
27.9.23	TK 967	0.5
27.9.23	TK 967	0.5

Apparatus Required for IS 10512



Double Jacket Funnel
With in & out circulation facility



Conical flask with
vacuum facility



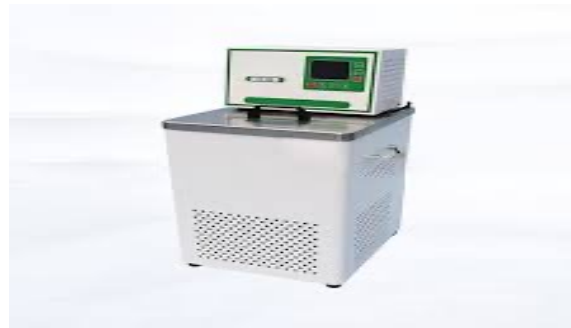
Circulation bath -20°C



Vacuum Pump > 5kPa



Steam bath



15±0.5°C



125±5°C



Balance ±0.5 mg

“Necessity is the Mother of Invention”



धन्यवाद