MOISTURE CONTENT OF BITUMEN EMULSIONS

(AN AZEOTROPIC DISTILLATION USING XYLENE)

Assemble the apparatus as shown above, following carefully the advise given concerning the use of all glass apparatus.

Shake the sample of emulsion to ensure thorough mixing as it tends to separate into 2 unmixable layers on standing for any length of time and weigh out 50g to 1d.p. into the Round bottom flask. This is easily achieved by zeroing the flask and carefully pouring the emulsion into the flask using a funnel held above the flask. Record the weight taken.

Add about 100 to 120ml of Xylene "Sulphur free" (CARE HARMFUL VAPOUR AND TOXIC BY SKIN CONTACT) to the emulsion and swirl the contents to ensure mixing of the 2 liquids. Add a spatula of Anti Bumping Crystals.

Turn on the water to the condenser, carefully (a sudden surge of water and air may break it) and gently increase flow to a maximum.

After fixing the flask to the apparatus, heat gently at first; slowly increase the temperature of the heating so as to produce a stable, steady boiling of the flasks contents. A mixture of water and Xylene distils over and collects in the bottom of the Dean and Stark apparatus:- Xylene on top.

If the rate of heating is too high, water and Xylene may be lost from the top of the condenser. Aim to have the "condensing ring" no more than $^{2}/_{3}$ the way up the water cooled section of the condenser.

The Xylene water mixture will settle out into 2 distinct layers but both will be slightly turbid due to minute drops of one solvent in the other. When the Xylene distils over clear, the water can be deemed to have been completely removed from the emulsion.

Allow the Xylene water to fully separate, using the spiral metal rod to dislodge any droplets of one liquid in the other and achieve complete separation.

Finally separate the water from the Xylene by opening the stop cock at the bottom of the column and only allowing the water to drain out; collecting it in a dry pre-weighed beaker.

The Xylene and bitumen solution left in the flask when cool may be collected via pre-marked "Winchester" bottle. It must not be poured down a sink and it must not be allowed to contaminate the Methylene Chloride still.

The Xylene may be separated from the jar by distillation and used again. Xylene is fairly expensive.

NOTES

1.Wash out all glass ware with "Methylene Chloride" as quickly as possible as emulsions tend to harden and "stick".

2.Carry out this procedure in a fume cupboard - Xylene is an arene and may posses carcinagenic properties.

NOTES ON THE USE OF "QUICK FIT" APPARATUS

Quick fit comes in a variety of sizes, not only volumes but also cone sizes and socket sizes. The cones and sockets of similar No's eg. 24/29 mate perfectly to provide a gas tight seal. It is however advisable to lubricate the cones slightly before insertion into a socket - but not too much grease. Special socket cone, "locks" are also to be found in the lab these more or less ensure complete locking of the joint whilst in use.

On making a joint - twist the cone gently around the socket until it grips - do not force it past this point otherwise the joint may freeze.

Don't wash glass jointed apparatus with chromic acid, or use a Phosphoric acid with ground glass joints - again permanent freezing may ensue.

Parting a frozen joint

This is not always successful but may sometimes be achieved by 2 methods.

1.Run hot water over the joint to attempt to expand the socket. As soon as water can be seen to ingress into the joint, it is free but if not gently twist and pull the joint to attempt to part the cone and socket.

2.Briskly rub string rapped once around the joint to again locally heat the cone and thus expand it. Again try to separate the joint at intervals.

If a frozen joint is successfully parted, wash the cone and socket in warm soapy water and apply a film of soft yellow petrol jelly after drying the components.

NEVER USE SOLUTIONS OF SODIUM HYDROXIDE IN QUICK FIT APPARATUS - NaOH dissolves glass quite readily and a permanent freeze will occur.