

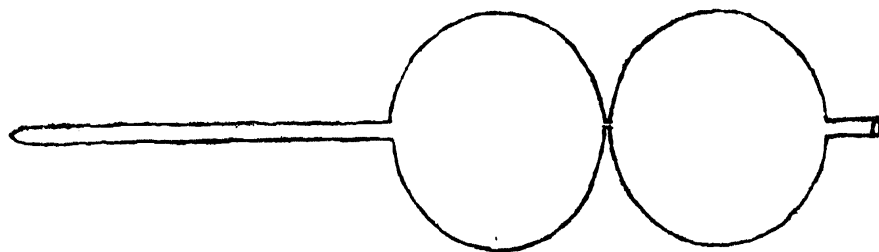
A BULB-TUBE AS A PYKNOMETER

At times it becomes inconvenient to employ the usual type of pyknometer for the determination of specific gravity of liquids which are available in small quantities or when a liquid is either volatile or highly viscous. U-type pyknometers of small capacity may sometimes be helpful. But the removal of air bubbles and the cleaning of the apparatus require special care. Adjustment of the column of the liquid to the given mark at the suction-end involves special effort.

These difficulties are practically overcome if a bulb-tube, as represented below, is used.

The overall length of this bulb-tube may be 6 cm. the nozzle-end alone being 3 cm. The nozzle has a constriction at the end. The two similar spherical bulbs are each 1 cm. in diameter, and connected by a very short and fine capillary tube.

The liquid under investigation is sucked in *via* the nozzle up to the capillary connecting the bulbs.



While weighing in the balance, the bulb-tube may be rested in the pan horizontally on a suitable stool made of metal wire. It is found that the liquid does not flow into the second empty bulb—not at least during the few minutes required for the weighing. A volatile liquid like ether can also be weighed in this apparatus without any appreciable loss due to evaporation at room temperature (25-30° C.). This is further ensured if the suction end is plugged after the liquid has been sucked in. Cleaning does not present a problem even in the case of a highly viscous liquid.

By this method the S.G. for ether is 0.6540 and glycerine 1.2560 at 26°.2, which compares favourably with standard values.

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THE 2:4 DINITRO-PHENYL HYDRAZINE COLOUR REACTION FOR VITAMIN K

Of the various colour reactions reported (reviewed by Reddy^{1,2} for vitamin K, the 2:4-dinitro-phenyl-hydrazine colour reaction described by Novelle³ was found to be the most useful because of the high sensitivity and stability of the colour developed. This reaction was found to be applicable to both fat-soluble and water-soluble vitamin K analogues that were available in the market. The following were the products examined:

The green colour was obtained with all these above compounds and the colour was found to be proportional to the concentration of the quinone.

Trade name	Manufacturer	Chemical nature
1 Kapilin (Injectule)	Glaxo Laboratories	2-Methyl 1 : 4-naphthaquinone (in oil)
2 Vitamindon K. (Tablets)	Indo-Pharma Pharmaceutical Works, Bombay	Methyl-naphthaquinone
3 Synkamin (Injectule)	Parke Davis & Co.	4-Amino-2-methyl-1-naphthol as hydrochloride (water-soluble)
4 Synkavit (Injectule)	Hoffman La Roche	2-Methyl-1 : 4-naphthohydroquinone diphosphoric ester as tetrasodium salt (water-soluble)

In adopting the technique of Novelle,³ we experienced great difficulty during the addition

of ammonia. Even the slightest excess of ammonia resulted in a reddish-yellow precipitate which masked the green colour. Further one cannot always be sure of the strength of the ammonia especially when the bottle has to be opened often. It was, therefore, considered necessary to study the reaction (especially the effect of pH) in detail and if possible to modify the method.

The green colour was obtained only when the final solution was definitely alkaline. At pH 6.8 a pale green colour was obtained with ammonia but the maximum intensity was obtained only beyond 7.3; but above 7.5 the use of ammonia resulted in a reddish-yellow precipitate. Various buffers and also NaOH in the place of ammonia were tried without success. Sodium carbonate, however, gave more satisfactory results. Even at high pH value Na₂CO₃ did not give rise to the reddish-yellow precipitate, and with this alkali the green colour was obtained only when the final solution was definitely alkaline to thymolphthalein. Further, the colour was quite stable even with excess of Na₂CO₃. Accordingly the following procedure was found to be satisfactory for the estimation of naphthaquinone derivatives, and hence vitamin K, using 2:4 dinitro-phenyl-hydrazine.

(i) Procedure for fat-soluble naphthaquinone derivatives (Kapilin and Vitamindon K): 0.5 to 0.15 mgm. of the substance (usually in oil) was shaken up with 0.5 c.c. of ethyl alcohol and to this 0.1 c.c. of a 1 per cent. solution of 2:4-dinitrophenyl-hydrazine in 2 N hydrochloric acid was added. It was then heated in a water-bath at 70° C. for about 10 minutes. It was then cooled, and 0.3 c.c. of a 20 per cent.