

NEW APPARATUS

A MELTING POINT APPARATUS OF BATH TYPE WITH MOTOR DRIVEN STIRRER

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FOR the determination of the melting point of readily powdered substances the British Pharmacopœia (Appendix IV) specifies an apparatus consisting of a glass heating vessel containing a suitable liquid, a suitable stirring device capable of rapidly mixing the liquid, a thermometer and capillary glass tubes of soft soda glass. In connection with a study of the effect of the type of capillary glass on melting point¹, an apparatus conforming to the basic requirements laid down in the Pharmacopœia, and having a motor-driven reciprocating stirrer, was constructed; with it concordant melting points were readily obtained. The apparatus now described, and developed from this earlier model for routine use, incorporates many new and useful features.

Figure 1 shows the general construction, the metal parts being mainly of brass and the base of wood. Oil is used as bath liquid and is circulated by an archimedean screw rotating at speeds up to 800 r.p.m., the oil being sucked in from the heated bottom region of the tube and discharged through the upper orifice of the pump housing. Up to three capillary melting point tubes may be inserted in the locating sleeves; the bottom ends, resting on the gauze just in front of the thermometer bulb, are illuminated by a beam of light through a hole at the side and observed through a window at the front of the draught screen. The bath is heated by a small Meker burner, the gas supply being controlled by a needle valve.

TABLE I
COMPARISON OF RESULTS OBTAINED USING TWO TYPES OF STIRRER AND EFFECT OF STIRRING SPEED ON MELTING POINT

Substance	Reciprocating stirrer	Archimedean screw stirrer (proposed apparatus)	
	M.pt. °C.	Speed of stirrer	M.pt. °C.
Maleic anhydride	51.9 52.0	Fast Medium Slow	51.8, 51.8 51.9, 52.0 52.0, 51.8
Benzoic acid	122.1 122.5	Fast Medium Slow	122.2, 122.2 122.0, 122.2 121.8, 121.8
Hydroquinone	173.0 173.2	Fast Medium Slow	173.0, 173.0 173.2, 173.2 172.6, 172.8
Sulphapyridine	191.8 191.6 192.0	Fast Medium Slow	192.0, 191.8 192.2, 192.0 192.0, 192.0 192.0

MELTING POINT APPARATUS OF BATH TYPE

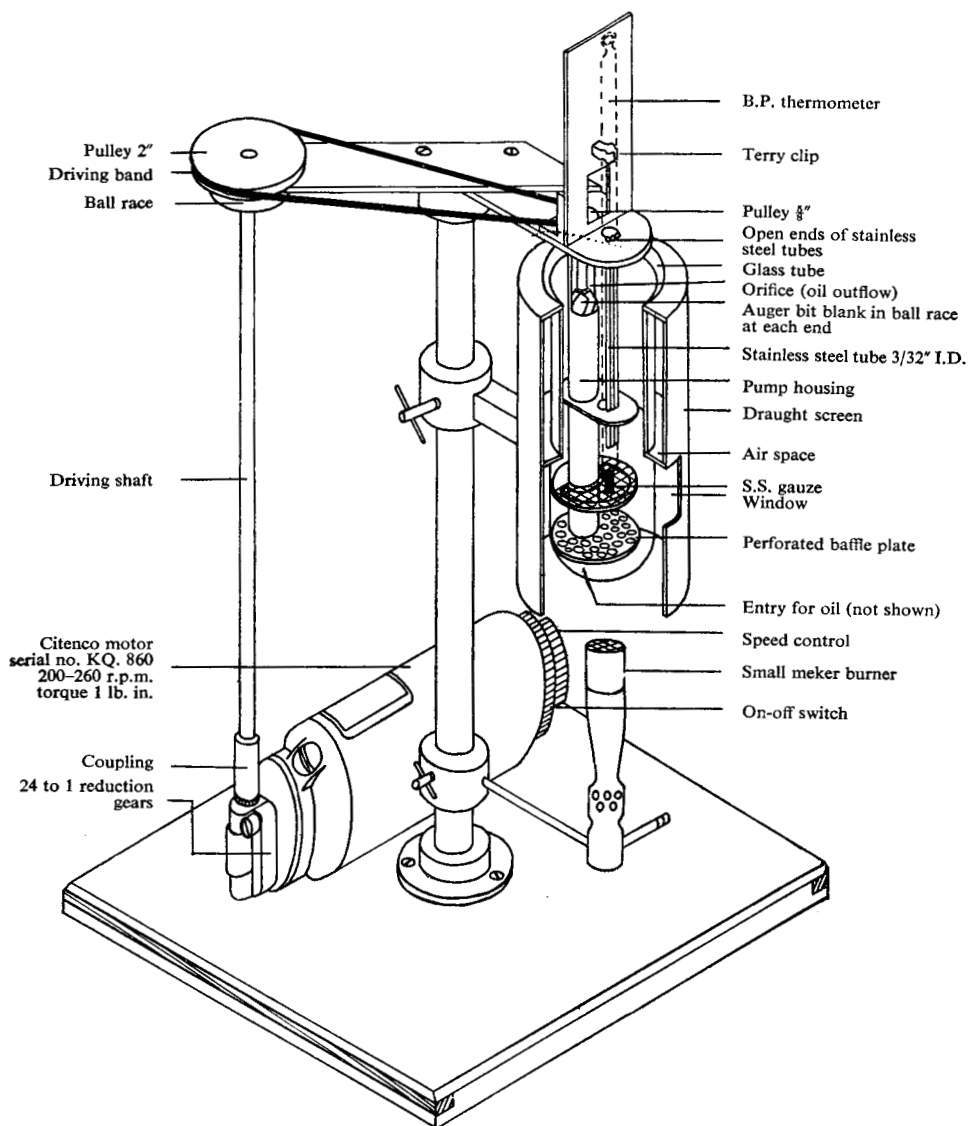


FIG. 1. Isometric diagram of melting point apparatus approximately one quarter actual size.

Melting point determinations on substances melting at temperatures ranging from 50° to 200° C. yielded concordant results in agreement with those obtained with the original reciprocating model (Table I). Stirring speed could be varied over a very wide range without effect on melting point.

Thanks are due to Miss S. Spice for the melting point determinations, to Mr. D. Hext for making the drawing, to Mr. E. D. Banks for suggesting

the design of the burner clamp and to the Directors of May and Baker, Ltd., for permission to publish this paper.

REFERENCE

1. Johnson and Ballard, *Quart. J. Pharm. Pharmacol.*, 1946, 19, 373.

LETTER TO THE EDITOR

Interaction between Chloroform and Ion Exchange Resin to give Carbon Monoxide

SIR,—“De-Acidite FF” is described as a unifunctional highly basic anion exchange resin and is stated to be stable towards all organic solvents and at temperatures up to 60° C. both in aqueous and non-aqueous media¹. According to Saunders² its functional group is quaternary ammonium and hence in its regenerated form the resin contains mobile hydroxyl ions¹.

In the course of experiments aimed at removing sulphonphthalein anions from chloroform solution by means of De-Acidite FF, the column having been first dehydrated with ethanol, the vigorous evolution of gas was observed; the gas was identified as carbon monoxide.

The hydrolysis of chloroform by aqueous sodium hydroxide to yield formate and the dehydration of formates by sulphuric acid to give carbon monoxide are well-known reactions; it is interesting to find both hydrolysis and dehydration occurring together in the presence of the ion exchange resin. This reaction is being studied in greater detail and possible analytical and preparative applications are being considered.

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REFERENCES

1. “Properties of, and Instructions for using Bead Resins, 6.” The Permutit Company Limited, p. 4.
2. Saunders, *J. Pharm. Pharmacol.*, 1953, 5, 569.

(ABSTRACTS *continued from p. 429*).

Racemorphan (Dromoran) Derivatives, Action and Addiction Liabilities of. H. Isbell and H. F. Fraser. (*J. Pharmacol.*, 1953, 107, 524.) Racemorphan, *dl*-3-hydroxy-*n*-methylnorphinan, is an analgesic drug with an addiction liability equal to morphine. Levorphan (the *l*-isomer) has previously been found to be active and dextrorphan (the *d*-isomer) inactive as analgesic and respiratory depressant. This paper reports the actions of dextrorphan and levorphan, and the methyl derivatives (dextromethorphan levomethorphan and racemethorphan), in human addicts to morphine. 3 to 4 mg. of levorphan, 20 to 30 mg. of racemethorphan and 10 to 20 mg. of levomethorphan injected subcutaneously into former morphine addicts were roughly equivalent to 30 mg. of morphine in causing euphoria. Effects on temperature, pulse rate and blood pressure were negligible, but respiration was significantly depressed. Levorphan and levomethorphan caused pupillary constriction, while codeine, dextrorphan and dextromethorphan did not. 40 to 60 mg. of racemethorphan orally relieved the withdrawal symptoms of morphine addicts. Levomethorphan was also effective, but dextrorphan and dextromethorphan were not. Both levorphan and levomethorphan had high addiction liabilities, while dextrorphan and dextromethorphan had not. The *l*-isomers therefore account for all the miotic, respiratory and addictive properties.

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