Molecular Crystals and Liquid Crystals
Publication details, including instructions for authors and subscription information:
http://www.tandfonline.com/loi/gmcl16

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Version of record first published: 29 Aug 2007.

To cite this article: Mary E. Neubert , Paul Norton & D. L. Fishel (1975): Using the Mettler FP-2 HOt Stage at Below-Room Temperatures, Molecular Crystals and Liquid Crystals, 31:3-4, 253-257

To link to this article: http://dx.doi.org/10.1080/15421407508082877

Mol. Cryst. Liq. Cryst., 1975, Vol. 31, pp. 253–257

Printed in Holland

Using the Mettler FP-2 Hot Stage at Below-Room Temperatures

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(*Received June 16, 1975*)

A modification of the Mettler FP-2 microscope hot-stage which provides for both rapid and controlled cooling below room temperature is described. Calibration of the stage was not affected by this modification. The use of sharp mesomorphic phase transition temperatures for calibration is also described.

Although the Mettler FP-2 microscope hot stage is used extensively by laboratories concerned with transition temperature measurements of mesomorphic compounds,[1–4] it does not work well in its standard configuration for measurements near or below room temperature. We have implemented a simple physical modification to allow nearly linear cooling to the lower limit of −20°C accommodated by the standard electronic temperature readout/control furnished with the FP-2. This was done by removing the small electric fan and replacing the standard tubing connector at the rear of the stage with a bulkhead Swagelok fitting which leads a short length of copper tubing to the side of the inner chamber containing the heating elements and sample slide mount. A small machined metal angle termination on the copper tubing is brazed to the side of the inner chamber so that a gas stream can be directed over the sample.

Externally, the Swagelok union was connected to a coiled length (to facilitate easy movement of the stage in a vertical direction) of insulated 1/8″ copper tubing which leads to a needle valve (Whitey #B-ORS2-A) that enables precise control of a stream of cooled high purity dry nitrogen gas via a coil immersed in a Dewar of either dry ice or liquid nitrogen. The necessary modifications are detailed in Figures 1 and 2.

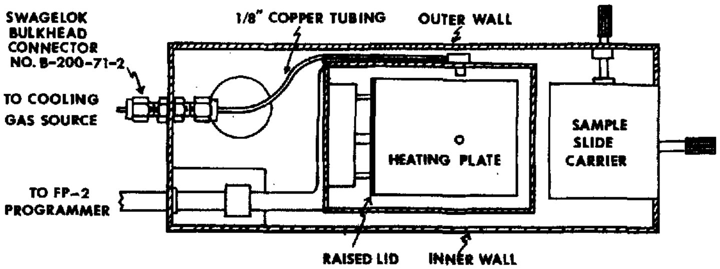

FIGURE 1 Top view of modified FP-2 hot stage. •

We experienced no problems with moisture condensation on optical parts even to −20° as long as a slow stream of gas was continuously allowed to flow.

The cooling rate can be controlled by adjustment of gas flow with the needle valve. The FP-2 programmer controls can be set to indicate when the desired rate is being exceeded. We are able to cool at the maximum rate of 10°/minute over the entire range down to −20°. This not only saves considerable time between runs but enabled us to reproducibly observe several monotropic transitions which are not evident at slower cooling rates. We have also discovered a number of new low temperature mesophases in compounds which did not show any such phases above room temperature.

A comparison of results on re-calibrating the stage after the modifications were completed with the previous calibration for the standard configuration suggests that the circulating fan previously used had little effect on the accuracy of measurements as shown in Table I (see Ref. 5 for an earlier report on calibration of the FP-2 stage).

Most standards recommended for calibrations of melting point equipment do not posses transitions which are as sharp and as well defined (or as

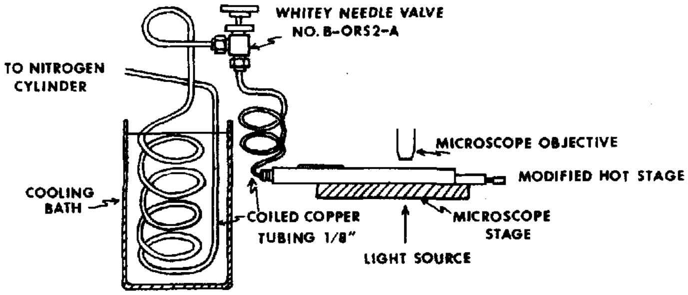

FIGURE 2 Cooling system attached to modified FP-2 hot stage.

readily repeatable) as are most mesophase–mesophase or mesophase–isotropic transitions. We feel that a properly chosen series of mesomorphic compounds could serve as better melting point standards.

Table II lists a series of commercially available (except for the phenyl benzoate) liquid crystals which we have studied as potential melting point standards. Unfortunately, most readily available mesomorphic compounds contain central groups such as the imine and azo functions which cause these compounds to be unstable over a period of time under normal laboratory conditions. In our studies, we found that many of these compounds did not give the sharp mesophase to isotropic transition temperatures that are found when they are pure.

TABLE I

Calibration data for modified FP-2 hot stage

Standard compound[a]	Melting points (°C) Reported	Unmodified stage	Modified stage[b]
3-Methoxy-4-hydroxybenzaldehyde (vanillin)	81–83	81.3–82.5	80.7–82.1
Acetanilide	114–116	114.2–115.2	113.4–114.9
4-Ethoxyacetanilide	134–136	132.3–135.2	133.3–135.4
4-Aminobenzenesulfonamide	164.5–166.5	163.1–165.8	162.4–165.4
N^1-2-Pyridylsulfanilamide	190–193	191.0–192.4	189.4–192.2
Caffeine	235–237.5	237.1–238.8	236.2–237.4

[a] Arthur H. Thomas melting point standards were used.

[b] Collected using the hot stage with a Leitz Orthulux Polarizing Microscope.

Two other problems were encountered in using these compounds as standards. Many of the higher melting materials tended to sublime causing fogging of the hot stage equipment and making observations difficult. Additionally, compounds which tend to have homeotropic phases make less desirable standards.

Table II lists those compounds for which we found a minimum of these problems. The crystal to mesophase transition range varied between 0.6 and 2.0° whereas the mesophase to isotropic range varied between 0.1 and 0.5°. We feel, however, that mesomorphic compounds which would be much better suited for melting point standards are the more stable aryl esters. A variety of these have been reported which have mesophase to isotropic transition temperatures over 0–300°, the range required to calibrate most melting point equipment.[1,6–10] We are currently studying such a series as potential melting point standards.

TABLE II

Potential mesomorphic melting point standards

Compound	$C \to M$[a]	$M \to I$[a]
4-*n*-Hexyloxyphenyl-4′-*n*-butylbenzoate[b]	30.7–31.3	48.5–48.6
4-Ethoxybenzylidene-4′-*n*-butylaniline[c]	35.1–36.5 (35.5[d], 35.3[e])	79.5–79.8 (79.0)[d,e]
4-(4′-Ethoxyphenylazo)phenyl Undecylenate	64.2–66.2 (62)[f]	108.6–108.9 (106)[f]
Diethyl 4,4′-azoxydibenzoate	113.8–114.7 (114)[f]	122.9–123.3 (122)[f]
4,4′-Azoxydiethoxybenzene (PAP)	134.9–136.8 (138)[f]	168.3–168.4 (168)[f]
4,4′-Anisalaminoazobenzene	169.6–170.9 (169)[f]	183.8–184.3 (184)[f]

[a] $C \to M$ respresents the crystal to mesophase transition temperature in °C. All mesophases reported here are nematic except for diethyl 4,4′-azoxydibenzoate which shows a smectic phase. $M \to I$ represents the mesophase to isotropic transition temperature. Temperatures in parentheses are literature values.

[b] The preparation of this compound was reported in Ref. 1.

[c] All remaining liquid crystals were obtained from Eastman Kodak and recrystallized once from abs. EtOH. It was found that a sharper $C \to M$ transition could be obtained if the crystals were first melted to the isotropic state, cooled until crystals were formed and reheated. This procedure gave a more homeogeneous layer of crystals.

[d] Ref. 11.

[e] Ref. 12.

[f] Ref. 13.

Acknowledgements

This work was supported by the National Science Foundation under Grants GH-34164X and DMR-74-13173.

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