

acetates and benzoates. These *colorless* compounds are therefore given the *lactoid* structure.

3. These hydroxy-tetrachlorofluorans, however, form *colored* sodium, ammonium and barium salts which are given the *quinoid* structure.

4. *2,4-Dibromo-3-hydroxy-tetrachlorofluoran*, and *2-bromo-3,4-dihydroxy-tetrachlorofluoran* have been prepared from the corresponding hydroxy-tetrachlorofluorans. They are *colorless* and form *colorless* acetates. However, they also are tautomeric, since they absorb ammonia gas and dissolve in alkalis with the development of color.

5. *3-Hydroxy-tetrachloro-hydrofluoranic acid* has been prepared by the reduction of the corresponding hydroxytetrachloro-fluoran. This acid and its acetate are *colorless* and dissolve in alkalis without development of color.

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[CONTRIBUTION FROM THE FIRESTONE TIRE AND RUBBER COMPANY]

AN IMPROVEMENT IN MACMULLIN'S AUTOMATIC APPARATUS FOR DETERMINING THE MELTING POINTS OF ORGANIC COMPOUNDS

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RECEIVED JUNE 3, 1926

PUBLISHED AUGUST 5, 1926

In studying MacMullin's description and operation of an automatic melting-point apparatus¹ it was concluded that any errors arising from lag in temperature could be eliminated by employing an automatic device that would remove the maximum thermometer from the bath at the instant the substance melted, and thereby improve the accuracy of the apparatus and decrease the time required for a determination.

With this idea in mind the MacMullin apparatus was modified in such a way that when the substance melted and shut off the heating circuit, it also started a motor which removed the thermometer from the bath. When the thermometer was withdrawn the motor was automatically shut off, thereby raising the thermometer to a definite height and eliminating any possibility of breaking it.

Apparatus.—Referring to Fig. 1, A represents the maximum thermometer, B the mercury bath, C the pulley over which the cord supporting the thermometer passes, D the point of attachment of the thermometer cord on Disk F (diameter = 10 cm.), E the point of attachment of the switch cord on Disk G (diameter = 5.6 cm.), H the motor (an ordinary fan motor, J the throw arm of the single-pole single-throw switch K, L the relay, M the 110-volt power line and N the connections to the coil posts of the relay of the MacMullin apparatus. The disks may be made of Beaver Board,

¹ MacMullin, *THIS JOURNAL*, **48**, 439 (1926).

G being on the side toward the motor. The cord from E to J is firmly attached on the circumference of G. D is an idler, so that if the motor should continue to run, the thermometer is merely raised and lowered. The thermometer is guided by passage through holes in two corks.

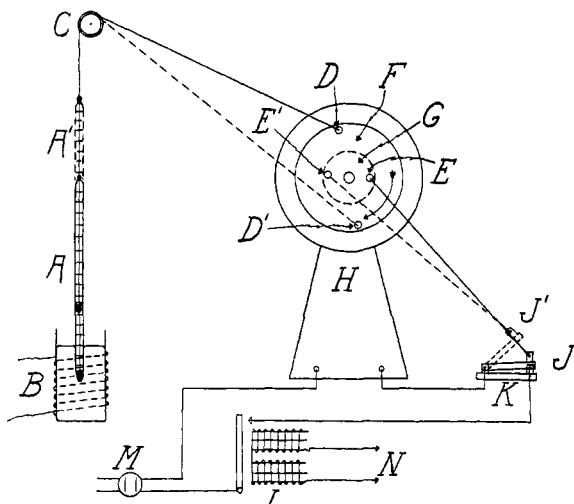


Fig. 1.

Method of Operation.—When the compound melts, contact is made through the relay L, thereby starting the motor which turns in the direction of the arrow. After the motor has made about a quarter revolu-

TABLE I
COMPARISON OF MELTING POINTS (°C.)

	MacMullin app.	Deviation from av.	Improved app.	Deviation from av.	Capillary tube	Inter. Crit. Tables (Vol. I)
<i>p</i> -Dichlorobenzene	55.3	+1.0	53.2	0	53.2	52.9
	54.5	+0.2	53.2	0	53.2	
	53.8	- .5	53.2	0		
	53.5	- .8	53.2	0		
	Av. 54.3		53.2		53.2	
Acetanilide	115.3	-0.3	114.8	-0.3	115	114.2
	116.1	+ .5	115.3	+ .2	115	
	116.0	+ .4	114.9	- .2		
	115.0	- .6	115.1	0		
	114.9	- .7	115.1	0		
	116.1	+ .5	115.1	0		
Av. 115.6		115.1		115		
Salicylic acid	161.0	+1.2	159.0	+0.1	159.4	159
	159.6	-0.2	158.6	- .3	159.2	
	158.8	-1.0	159.0	+ .1	159.3	
	Av. 159.8		158.9		159.3	

tion the cord from E to J opens the switch, thereby breaking the contact in the power line, and the pull on the switch tends to act as a brake to stop the motor in the position indicated by the primed letters. D has thereby moved to D' and in so doing has withdrawn the thermometer to a position about 5 cm. above the surface of the bath. It was found by several trials that the maximum point registered on the thermometer was not altered by this method of removal from the bath. The table shows the relationship between melting points obtained with the MacMullin apparatus and with the improved apparatus. The temperatures given are all corrected and the same thermometer was used in all cases.

From Table I it is readily seen that the results obtained with the improved apparatus are by far the more consistent and are nearer to the correct readings as determined by the capillary-tube method.

Different rates of heating produce different temperature lags in the MacMullin apparatus, whereas the results obtained by the improved method are practically independent of the rate of heating. One example will suffice to show these differences.

Wattage	Temp. rise °C. per min.	Melting points (°C.)	
		MacMullin app.	Improved app.
115	1.5	122.7	122.4
175	8	123.8	122.7

Calibration of the apparatus for radiation and rate of heating is therefore unnecessary and less time is consumed for each determination, an item which is of particular interest to one who has several melting points to determine.

Summary

An improvement in an automatic melting point apparatus for organic compounds has been described.

It has been shown that the precision obtained with this improvement is comparable to that obtained by the capillary tube method.

It has also been shown that the results obtained are practically independent of the rate of heating and that calibration of the apparatus is therefore unnecessary.

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