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A Review on the Determination of Melting Point Measurement System

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ABSTRACT: A melting point apparatus is a scientific instrument which is used to determine the melting point of a chemical substance. A melting point can be used to identify the purity of a chemical substance. The melting point of a solid is the temperature at which the solid exists in equilibrium with its liquid state under an external pressure of one atmosphere. The melting point range is defined as the interval between the beginning of liquidification and completion of liquidification process. A pure crystalline organic chemical compound usually possesses a sharp melting point and it melts completely over a narrow range of temperature which is not more than 0.5-1.0^{o}C, provided good technique is followed. The presence of even small amounts of impurities in the chemical compound usually produces a change in the melting point range. The present work gives the earlier, current developmental status and design considerations of existing melting point determining instruments with optical arrangement, analysis, calibrating chemical compounds and techniques used for automation. The above said areas are covered in the review. The intention of the present work is to develop the future research work in the field of melting point determination systems.

KEYWORDS: melting point, calibrating chemical compounds, optical arrangement.

I.INTRODUCTION

The melting point of a solid is the temperature at which it changes state from solid to liquid at atmospheric pressure. The melting point of a substance depends upon the pressure which is usually termed as standard pressure. If the temperature is said to be decreased we can observe a change from liquid to solid, only in some chemical compounds which is known as the freezing point or crystallization point. The freezing point is not considered as a characteristic property of a chemical substance. Usually, the Melting point of a chemical substance is mentioned in Melting point range of 0-3.0^{o}C depending upon the purity of a given substance. The melting-point apparatus can be used in pharmaceutical industry, chemical industry, textile industry, dyestuff. The price of commercially available Melting Point Apparatus varies from 3 Thousands to 10 Lakh INR depending upon the techniques and features embedded.

II.LITERATURE REVIEW

The Belgian physical chemist, Jean Timmermans, in his classic monograph, On the Concept of Species in Chemistry, attributed the first use of melting points as a means of characterizing organic compounds to the famous 1832 paper by the German chemists, Justus von Liebig and Friedrich Wohler, on the chemistry of the benzoyl radical in which they reported the melting point of benzamide [1, 2].
The first generation of melting point measuring system is as shown in the figure 1which used by Gattermann [3]. In this method the melting point capillary with a chemical compound is attached to the stem of the thermometer which is suspended in a long-necked, round-bottom flask filled with concentrated sulfuric acid or some other liquid paraffin with a high boiling point and is carefully heated using a Bunsen burner.

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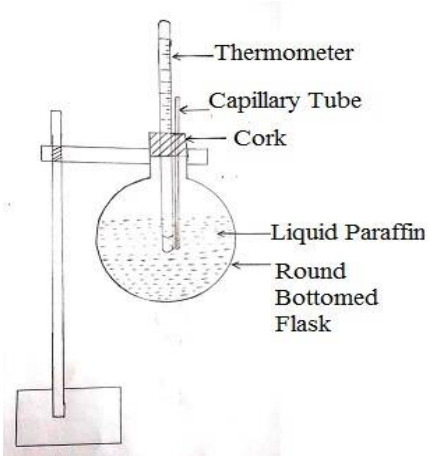

Figure 1. Simple melting-point apparatus used by Gattermann in 1894

The second generation started with the "Thiele melting-point tube," which looks like a tube of "b" shape as show in figure 2. In this system the Bunsen burner is kept to one side of the tube not directly beneath the thermometer. The capillary is suspended to the stem of the thermometer [4].

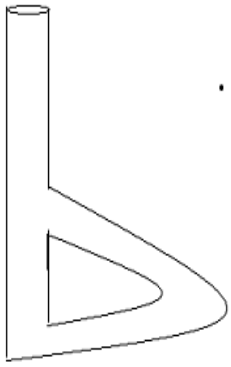

Figure 2: Thiele Melting point tube

The third generation started with the electrical heating of the system by passing appropriate voltage to generate the heat required.

Mean while in 1961 a patent has been filed by Sidney Kasman regarding to the melting point measuring system which consists of electrically heated melting block fitted with V-Tube assembly [5].

The fourth generation of the system shown in the figure 3 uses a metal block with a heating element which is heated with appropriate range by passing voltage through the rheostat or variac. Here the capillary with chemical compound is inserted in one hole and the thermometer in the another hole provided to the block. A glass window is equipped to observe the internal changes happening in the capillary tube with an eye piece. Due to in expensive this system is usually still used in colleges and laboratories.

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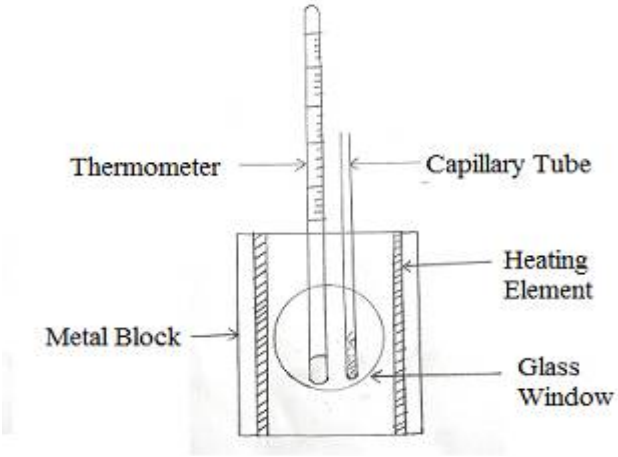

Figure 3: Widely used system in colleges

The main aim of this paper is

- to deliver an outline of the Melting Point Measuring Systems
- Design and development of the experimental setup for determination of melting point

Principle of commercially available Instruments for melting point determination over the course of years is presented in the table 1.

Table :1

Year	Medium	Determination type
1957 -1974	Silicon oil Thermometer	Visual
1974 – 1996	Metal block with Thermometer	Visual
1996 – 2009	Metal block with Pt-100 resistor	Visual and manual Transmission
2009 – now	Metal block with Pt-100 resistor	Visual and manual Reflection with image processing

The Melting Point Apparatus has to be regularly calibrated with certain known melting point range of chemical compounds. Eight USP Standard Chemical compounds which are used for calibration of Melting Point Apparatus are presented in the table2.

Table:2

S.No	USP Standard	Lot Nr.	Max. limit of the melting range acceptance*
1	Phenyl salicylate	F1M306	< 1.8 °C between 40.4 – 44.3 °C
2	Vanillin	L0M294	< 2.1 °C between 80.3 – 84.2 °C
3	Acetanilide	M1M285	< 2.0 °C between 113.2 – 116.8 °C
4	Phenacetin	J1J346	< 2.0 °C between 133.8 – 136.9 °C
5	Sulfanilamide	R007N0	< 2.2 °C between 163.7 – 167.2 °C
6	Sulfapyridine	K0L179	< 2.3 °C between 189.1 – 193.4 °C
7	Caffeine	K2M218	< 1.0 °C between 235.2 – 238.1 °C
8	Theophylline	F1M305	< 2.1 °C between 270.3 – 274.2 °C

Automatic determination of melting point range in modern melting point determination instruments: The pictorial representation of an instrument used for automatic and visual determination is represented in the figure 4.

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The capillary tube containing sample is placed metal block made up of aluminum, which is electrically heated by means of a heating element and further the temperature is controlled by an electronic circuitry. Measurement of temperature is done by using RTD- Pt100/ PT-500 with an appropriate signal conditioning circuit. The heating block can be maintained accurately at an user defined temperature by the heating element control circuit and can be heated at a prescribed rate. A source of LED light illuminates the glass capillary tubes containing sample from the front side. A built-in camera continuously captures the reflected real-time images of the illuminated sample and determines the melting point from the analysis of stored images. Melting point is detected by reflection with image processing. Figure 5 represents the block diagram of a sophisticated system which may be equipped by a microcontroller along with a touch screen to input data and graphical display to display the images.

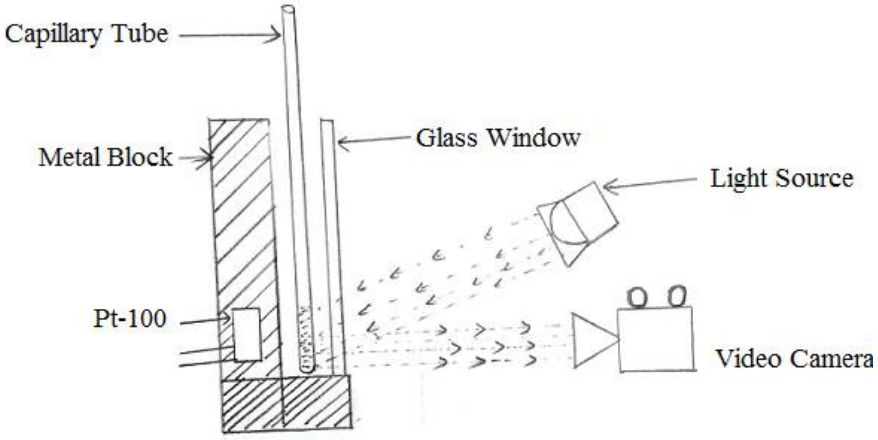

Figure 4: Automatic detection of melting point by measuring reflected light.

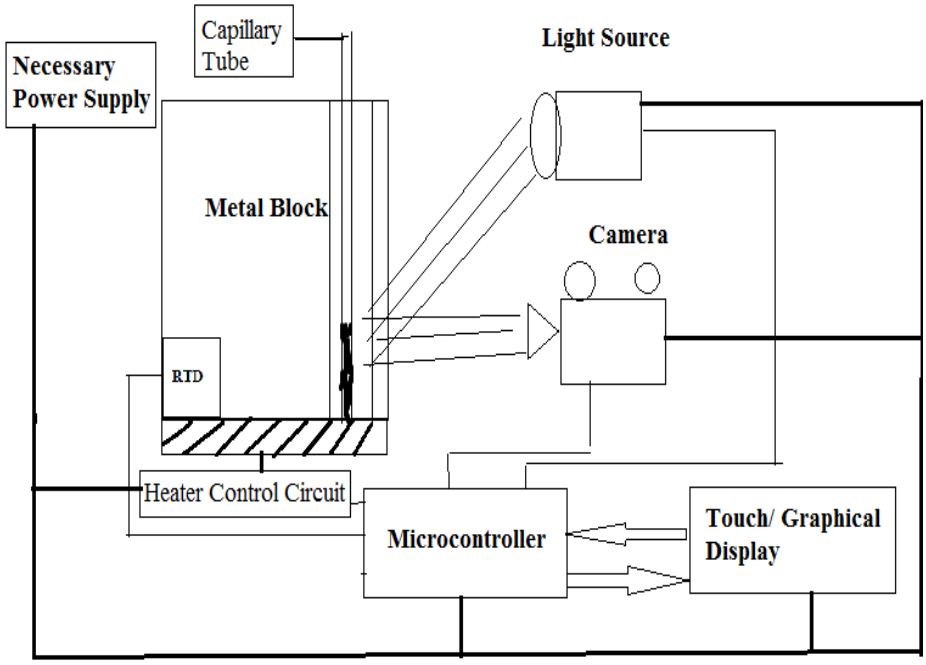

Figure 4: Modern automated melting point measuring system

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III. CONCLUSION

The present work can provide good research knowledge on melting point measuring systems with experimental methods. The hardware and software implementation provides to develop new dimensions in the field of Thermal behavior of chemical compounds. Therefore, the paper provides an overview for a researcher interested in the field of Thermal Analysis of chemical compounds.

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BIOGRAPHY

C. Sandeep Kumar Reddy completed M.Sc Electronics and Instrumentation from Sri Krishnadevaraya University in 2009. He is pursuing Ph.D in Instrumentation. His areas of interest in Instrumentation are Scientific and Analytical Instrumentation

Dr. K.K. Azam Khan, Scientist, EchoMRI Corporation Pte Ltd, Singapore. He completed Ph. D in Instrumentation in Sri Krishnadevaraya University. He has joined as a Development Engineer at ELICO Hyderabad, in 1992 and worked there for a period of 1 year. He has joined as a faculty member in the Department of Instrumentation as an Assistant Professor for a period of 8 years. He has joined as a faculty member Temasek Engineering School, Singapore and worked there for a period of 10 years. His areas of interest are Bio-Medical Instrumentation, Scientific and Analytical Instrumentation. He is also well versed with Embedded Microcontrollers and Programming.

Prof.C.Nagaraja, Professor, Department of Instrumentation Sri Krishnadevaraya University. He completed his M.Sc, M.Phil, Ph.D., in Instrumentation from the same University. He has joined as the faculty of Instrumentation & USIC in 1993, Sri Krishnadevaraya University as an Assistant Professor. He is a Fellow Member of Institute of Electronics and Communication Engineers (New Delhi). He completed a major research project sponsored by UGC on "Design and development of Microcontroller based low cost pulse oximeter". He has organized 3 National workshops and attend 3 National symposium conducted by Instrument society of India. He was the resource person for several workshops and seminars in instrumentation organized by Sri Krishnadevaraya University. He is a life member of Instrument society of India, Bangalore. He has published nearly 20 papers in national and international journals in Instrumentation. His areas of specializations are Bio-medical instrumentation, Analytical Instrumentation, Microcontroller based application development