HALLIKAINEN Instruments
INDUSTRIAL and SCIENTIFIC

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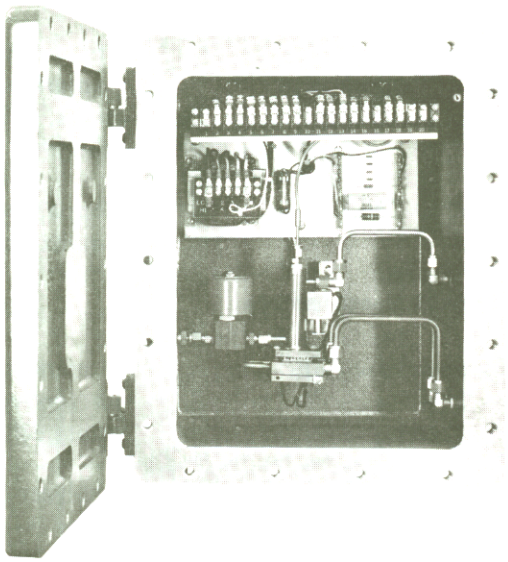
FREEZING POINT ANALYZER

MODEL 1470

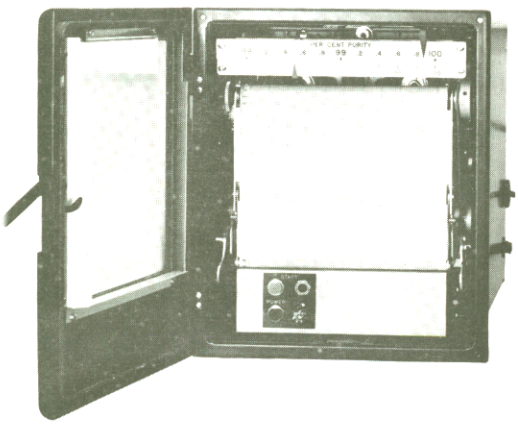
Introduction

The Model 1470 Freezing Point Analyzer is a continuous process analyzer that measures the freezing point of liquids as a means of purity determination. On-stream analysis eliminates laboratory time and cost of performing purity tests by the standard ASTM D 1015-55 method. Correction of off-specification products can be made manually or through closed control loops obviating the necessity of holding tanks. The purity specification margin can be held closer with a corresponding increase in yield. A liquid sample is supercooled below its freezing point and then caused to partially fuse into a solid by mechanical impact. The exact freezing point temperature is indicated and recorded by a two-pen strip chart recorder. This method is simpler and more reliable than the comparable ASTM test. Stirring of the sample during cooldown is not required.



Applications

Determination of purity is applicable to essentially pure compounds such as benzene, toluene, ethylbenzene, o-xylene, p-xylene and probably many other compounds with known freezing points for zero impurity and cryoscopic constants. The samples must be water-free and capable of being supercooled. Corrosive solvents may require a special sample cell (consult factory).



Operation

A representative dry sample is introduced into the copper sample cell through a timer-controlled solenoid valve. The valve is opened for approximately 30 seconds to purge the cell of the previous sample and to raise the cell temperature to the required starting level. A thermoelectric cooler is energized to cool the sample at a rate of about 6°C. per minute. As cooling progresses, the blue and red pens of the two-pen recorder move downscale to the left as the sample temperature is sensed by a thermistor centrally immersed in the cell. When both have reached the left full scale position, which represents the required amount of supercooling, a knocker solenoid is energized which mechanically shocks the sample into freezing. The frozen material along the walls of the cell thermally insulates the liquid center core. This central portion of the sample remains at the freezing temperature for several minutes. The freezing point temperature, and thus the percent purity, is indicated and recorded by the red pen of the recorder which is calibrated in the range of 98% to 100% purity, or as required. When the display period is completed, the programmer automatically opens the solenoid valve to flush the cell with fresh, warm sample and the cycle repeats. The time of one cycle can vary depending on the product being measured but several tests per hour can normally be expected.

General Description

The instrument is composed of two separate units; analyzer and recorder. An explosion-proof box houses the sample cell, thermo-electric cooler, power supply and water-cooled heat sink, solenoid valve, knocker solenoid and thermistor temperature detector plus the electronic control chassis. This unit should be located close to the main process stream from which the sample is to be drawn.

The recorder is a modified Bristol 2-pen recorder and is usually mounted in a control house remote to the sample point. Included in the recorder case are the timer-programmer, pen-controlled switches, and thermistor bridge circuits.

Description of Components

Housings	The housing for the sample cell, thermoelectric cooler and electronics is an explosion-proof enclosure designed for use in Class 1, Group D, Division 1 areas. The recorder case is not explosion-proof and is designated for installation in a control house.
Sample Cell	Copper with 15 ml volume (standard) or copper with s.s. liner for corrosive liquids (at extra cost).

General Specifications

Sample flow rate:	Maximum possible in sweepstream. Total flow through sample cell depends on product. With p-xylene, sample must be 70°F. and dry. Pressure drop across cell is approximately 1 to 2 psi with 30-second flushing period.
Installation:	Analyzer mounted in vibration-free area near main process stream. Cooling water is required for thermoelectric cooler heat sink.
Maximum line pressure:	200 psi.
Cycle frequency:	Varies with product. Approximately 11 minutes for p-xylene.
Cooling rate:	Varies with product, about 6°C./min. for p-xylene.
Supercool:	Varies with product, 5°C. below freezing point for p-xylene.
Cooling water:	5-10 gph at 65-80°F.
Utilities:	115 volt, 60 cycle, single phase
Thermal cutout:	Thermoswitch deenergizes cooler if module rises above 135°F. due to loss of or inadequate flow of cooling water.
Span:	As needed depending upon product. 0.8°C. (red pen) for 98-100% purity of of p-xylene (100% = 13.26°C.) For benzene, 0.5°C. corresponds to 99%-100% purity (100% = 5.53°C.) Phthalic anhydride, 1°C. corresponds to 98% - 100% purity (100% = 131.1°C.)
Repeatability:	Better than ± 0.004°C. with p-xylene.