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Pressure Chamber for Use During Differential Thermal Analyses

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Synopsis

This article describes a simple cell suitable for use with the Du Pont 900 differential thermal analyzer to maintain pressures up to 150 psi. Also discussed are two applications of such a device.

Introduction

The Du Pont 900 differential thermal analyzer (DTA), can be used in controlled atmospheres up to one atmosphere. One of the applications for the DTA in this laboratory was in the measurement of a property known as shrinkage temperature in hides and leather specimens immersed in water.[1] However, some chrome and other polytan leather specimens undergo shrinkage in a water medium at temperatures above the boiling point of water, 100°C. The development of the pressure device described in this article was necessary to undertake such a study.

Construction and Operation

Basically the cell, as shown schematically in Figure 1, is composed of a pressure chamber mounted above a base plate containing an electric plug permitting attachment to the Du Pont 900 DTA. The pressure cell was constructed with the same internal design as a typical Du Pont DTA cell; i.e., aluminum cell block with three wells for thermocouples, one to control temperature programming and two others to monitor sample and reference temperatures, with a heater well symmetrical with the others. The cell block with all its components was placed in a marinite holder and mounted to the bottom of the chamber on glass legs in such a fashion that an air void existed. The space insured even heat transfer to the thermocouple system. The construction of the chamber is steel with a paper-phenolic core through which 24 gauge chromel-alumel thermocouple wire is run to act as a conducting medium between the cell assembly and the pin of the instrument console. Nitrogen or other gas supplies the pressure which is

read out on a gage mounted to the top of the chamber. The cell was built to hold up to 150 psi. A calibration of the cell in the pressure device was performed. NBS samples of various crystalline compounds were run in

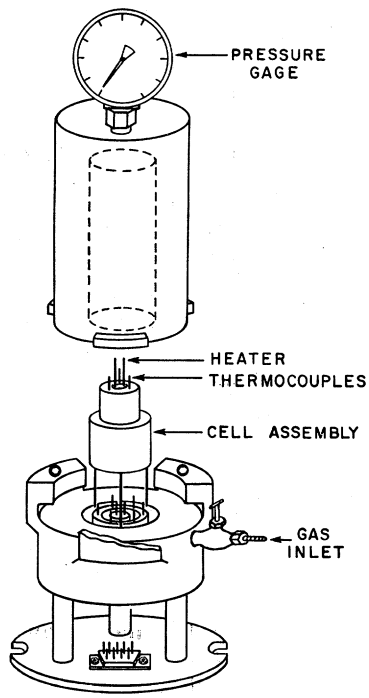

Fig. 1. Pressure device for DuPont 900 differential thermal analyzer.

the pressure cell and then in the regular DTA cell. The reproducibility of the DTA thermograms for samples run in the pressure and regular DTA cells was well within the experimental error of the instrument, ±0.25°C.

Performance

As was stated previously, the specific use for which this pressure chamber was developed was to maintain a pressure such that the boiling of water would not take place when measurements of shrinkage temperatures of hides and leathers, occurring above 95°C., were being made in a water medium. The resulting thermograms were of the same shape as comparable leather–water specimens which had their transitions below 100°C.[1] Figure 2 shows a typical thermogram on a chrome-tanned hide immersed in water. The shrinkage temperature results obtained by use of DTA compared favorably with those results obtained with a visual free end pressure device for fifteen different types of leather.[2]

Another application for pressure DTA is in the determination of the dependence of the vapor pressure on temperature in a given system by use

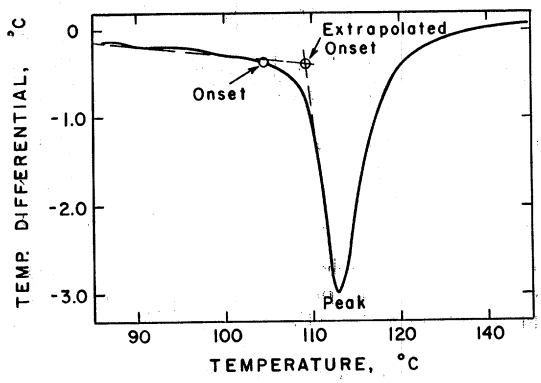

Fig. 2. Typical DTA thermogram for a chrome-tanned leather immersed in water.

of the Clausius-Clapeyron equation. The integrated form of the equation is

$$\ln p = -(\Delta H_V/R)(1/T) + C$$

where p is vapor pressure (in atmospheres), ΔH_V is the heat of vaporation (in calories/mole), R is the gas constant (1.987 cal./°K./mole), T is absolute temperature, and C is a constant related to the entropy of the transition. With the pressure device described, this determination was made quite readily. Shown in Table I are the DTA peak temperatures for the vaporization of water at three different pressures above atmospheric.

TABLE I
Transformation Temperature and Vapor Pressure of Water

Total pressure, mm.	DTA peak temperature, °C.	Predicted boiling point, °C.[a]
978.6	107.2	107.3
1275.0	115.1	115.2
1659.0	123.5	123.5

[a] Data of *Handbook of Chemistry and Physics.*[3]

A least-square calculation of the above data yields a heat of vaporization of 9.55 kcal./mole. The predicted value between 107 and 124°C. is 9.47 kcal./mole.

Summary

A simple device was developed and tested which allows measurement of thermal behavior by DTA at elevated pressures.

Mention of specific firms and products does not imply endorsement by the U. S. Department of Agriculture over others of a similar nature.

References

1. Witnauer, L. P., and A. Wisnewski, *J. Am. Leather Chemists' Assoc.*, **59**, 598 (1964).
2. Naghski, J., A. Wisnewski, E. Harris, and L. P. Witnauer, *J. Am. Leather Chemists' Assoc.*, in press.
3. *Handbook of Chemistry and Physics*, 45th ed., Chemical Rubber Publishing Co., Cleveland (1964).

Résumé

Cet article decrit une cellule simple adaptable à l'analyseur thermique differentiel Du Pont 900 en vue de maintenir les pressions jusqu'à 150 livres/pouces^2. On discute également deux applications de cette méthode.

Zusammenfassung

In der vorliegenden Arbeit wird eine einfache Zelle beschrieben, die zur Verwendung mit dem Du Pont 900 Differentialthermoanalysator zur Aufrechterhaltung von Drucken bis zu 150 lbs/in^2 geeignet ist. Zwei Anwendungen einer solchen Vorrichtung werden diskutiert.

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