

NOTE

Apparatus for Determination of Moisture Content of Solids and the Sorption of Gases and Vapors by Solids at Elevated Temperatures.—In the course of an investigation conducted by the writer it was desirable to determine not only the water content but the quantity of water vapor sorbed by a solid, granular substance from various steam-air mixtures at temperatures from 100 to 450°. The apparatus developed for this purpose was unique in several respects and, although requiring the services of a skilled glass blower, may find application in work of a similar nature by others.

The apparatus, shown in the accompanying sketch, consists essentially of a constant-temperature vapor-bath B with liquid boiling under automatically controlled reduced pressure,¹ an air-bath M and a detachable weighing tube D. The construction of the apparatus will be apparent from a description of the procedure in determining, for example, the sorption of water vapor. The method consists in passing a steam-air mixture² over a weighed amount of the solid material at constant temperature until equilibrium is established, as evidenced by no further increase in weight. The steam-air mixture enters the air-bath tube M, which is covered by the ground glass cap F, through the preheating coil J. The glass stopper G hanging from the hook on the glass rod H, allows the gases to escape through the tube D containing the granular material and thence out through the ground-glass joint E which also serves as a support for the tube D. The tube M is sealed at L to the inner tube N of the vapor-bath so that the entire cap F is about two inches below the top of the vapor-bath. The vapor from the boiling liquid³ in B then fills the annular space between the outer wall of B and the inner tube N, thus maintaining the cap at the bath temperature. The temperature of the system may be read from the mercury thermometer suspended in the vapor-bath from the ground-glass stopper as shown. The outlet tube above the cap is wound with nichrome wire and heated electrically to prevent condensation of steam. A loose plug of glass or cotton wool in the open end of N excludes air currents cooling the cap F.

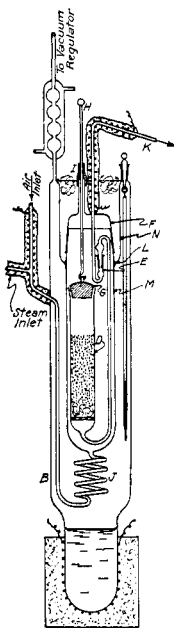


Fig. 1.

The experiment may be interrupted at any time for the purpose of weigh-

¹ For description of automatic control device see Kuentzel, *THIS JOURNAL*, **51**, 3347 (1929).

² See Kuentzel, "The Preferential Catalytic Oxidation of Carbon Monoxide in the Presence of Hydrogen," Part I, for description of a suitable steam generator.

³ A list of suitable liquids and their boiling points is given in "International Critical Tables," 1926, Vol. I, p. 66.

ing by sliding the rod H through the rubber connector I, thus closing the stopper G. The tube K is then closed by a short rubber tube and glass bead and the entire sorption tube and cap ensemble removed and placed in a desiccator to cool. A vacuum type desiccator carrying a bottle neck in its cover enables one to insert the weighing tube into the desiccator in a vertical position. A thin rubber sleeve attached to the bottle neck, into which the cap F fits, then seals the desiccator. When cool, the small tube D is detached at the ground joint E for weighing. A correction must, of course, be made for the steam that condenses in the free space of the tube upon cooling.

When the apparatus is used for determining the moisture content of solid materials, the steam generator is cut off and a stream of completely dried air or nitrogen passed over the material in D. The effluent gases are then passed through a series of weighed drying tubes attached at K to collect the moisture. The loss in weight of the material in D serves as a check. The dissociation of moist materials which decompose upon heating with evolution of gases not absorbable by the drying agents used may thus readily be obtained by determining the total loss in weight of the material itself as well as the amount of water vapor evolved. The ability to interrupt the experiment easily and to determine the change in weight also enables one to follow the *rate* of sorption, drying or dissociation quite readily.

CONTRIBUTION FROM
FERTILIZER AND FIXED NITROGEN INVESTIGATIONS
BUREAU OF CHEMISTRY AND SOILS
DEPARTMENT OF AGRICULTURE
WASHINGTON, D. C.
RECEIVED JUNE 12, 1929
PUBLISHED DECEMBER 11, 1929

WARD E. KUENTZEL

[CONTRIBUTION FROM THE CHEMICAL LABORATORY OF NORTHWESTERN UNIVERSITY]

THE PYROLYSIS OF HYDROCARBONS: ISOBUTYLENE¹

BY CHARLES D. HURD² AND L. U. SPENCE³

RECEIVED MAY 20, 1929

PUBLISHED DECEMBER 11, 1929

In the first paper⁴ of this series, data were recorded for the pyrolysis of *n*-butane and *isobutane*. The present paper concerns itself with *isobutylene*, a hydrocarbon which possesses a branched chain of four carbons,

¹ This paper contains results obtained in an investigation on "The Non-Catalytic Thermal Decomposition of Pure Hydrocarbons and Related Compounds," listed as Project No. 18 of American Petroleum Institute Research. Financial assistance in this work has been received from a research fund of the American Petroleum Institute donated by the Universal Oil Products Company. This fund is being administered by the Institute with the cooperation of the Central Petroleum Committee of the National Research Council.

² Director, Project No. 18.

³ American Petroleum Institute Junior Research Fellow.

⁴ Hurd and Spence, *THIS JOURNAL*, **51**, 3353 (1929).