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Note

A New Method for the Determination of Water Content in Extra Dry Gases

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The water content in extra dry gases can be determined by the condensation of water vapor on the cooled walls of the container. After separation, vapor pressure is measured at room temperature by the Pirani vacuummeter. In favorable conditions water content as low as $1 \mu\text{g}$ per liter of gas at NTP can be detected.

The described method is based on the separation of water from the gas sample by cooling it to -100°C and pumping off the dried gas. After heating the vessel back to room temperature, the residual pressure determined by the Pirani vacuummeter is equal to the partial pressure of the water initially presented in the sample.

The experimental arrangement is given in Fig. 1. Through a cylindrical glass vessel, of about 1 liter in volume, a moderate flow of gas along the direction A, B, C is maintained for some minutes in order to assure the representativity of the sample. After closing the valves B and C the isolated sample is surrounded by gasoline or ethyl alcohol kept at -100°C approximately. After 5 min of cooling time, the gas is pumped off through open valves

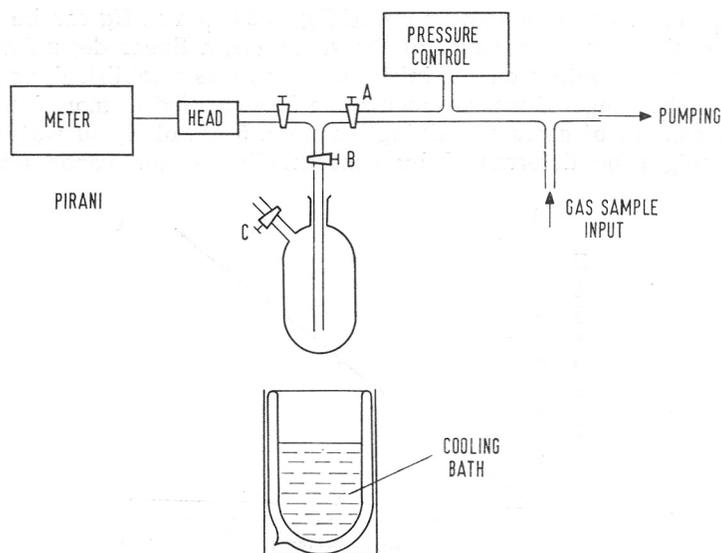


Fig. 1. Apparatus for the determination of water content.

A and B by the rotary and oil diffusion pumps until a negligible pressure is indicated by the Pirani vacuum gauge. The evacuated vessel is heated to the initial gas temperature and the residual pressure is determined by the Pirani vacuum gauge. As the employed Pirani gauge Edwards M6A was originally calibrated for air its sensitivity to water vapor had to be examined. The saturated vapor pressure of ice¹ under equilibrium condition was utilized for calibration. For this purpose the sensing head was connected to the vessel with ice at its equilibrium vapor pressure. By varying the vessel's temperature from -20°C to -70°C while the sensing head was held at room temperature, the full range of Pirani gauge was covered. From Fig. 2, where the Pirani

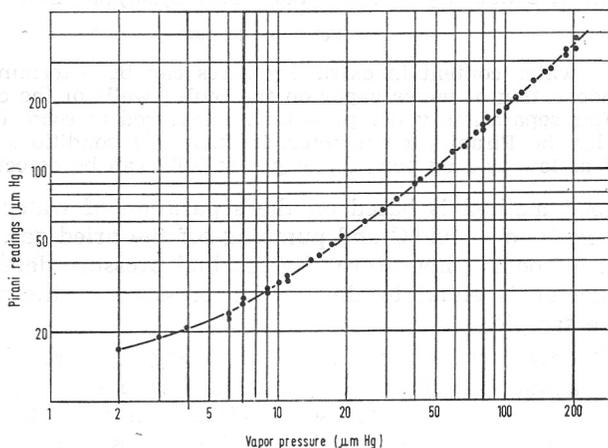


Fig. 2. Plot of Pirani readings *versus* vapor pressure of water.

readings *versus* corresponding vapor pressure are plotted, it is evident that water vapor pressure from about $5\ \mu\text{m}$ of Hg to $200\ \mu\text{m}$ of Hg can be determined. By analysing several samples of the same gas, a linear dependence of the determined water content on the initial pressure was established, as shown on Fig. 3. Thus the range of measurements can be extended to more humid gases. The water content of gases containing less than $5\ \mu\text{m}$ of Hg of water pressure at 1 atm might be determined by concentrating water vapor from larger samples.

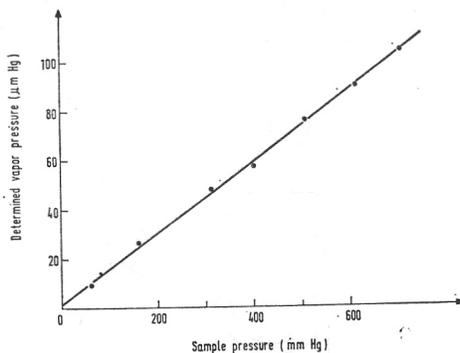


Fig. 3. Dependence of determined water content *versus* initial sample pressure.

The adsorption of water molecules to the vessel walls does not represent a serious limitation. While a monomolecular layer of molecules adsorbed inside a spherical vessel with a volume of about 1 liter would correspond to the pressure of 20 μm of Hg, a more elaborate estimation of the equilibrium between the adsorbed film and gaseous phase^{2,3} shows that only 10^{-10} of the adsorptive centers at room temperature are filled. The uncertainty due to the adsorption can thus be overlooked.

In a set of experiments it was confirmed that a 5 minutes cooling was sufficient as no measurable increase of residual pressure was detected after prolonged cooling. Additionally, only traces of water were detected in the gas pumped from the vessel after the cooling period. Besides, the method was tested for evaporation of water from ice frozen on the walls during the pumping period. No differences in residual water pressure were obtained by varying the pumping time from 5 minutes to 1 hour.

The described method can be used for the determination of the water content in the majority of technical gases such as hydrogen, helium, oxygen, nitrogen, argon and others with sufficiently low boiling points. In comparison with other methods suitable for measurements in this range^{4,5} the proposed method requires no calibration with gas samples of known humidity.

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IZVLEČEK

Nova metoda za določevanje vode v zelo suhih plinih

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Vlaga v plinu se meri s pomočjo separacije z ohlajenjem plinskega vzorca v stekleni cilindrični posodi do temperature -100°C . Po 5 minutnem hlajenju se iz posode odčrpa osušeni plin, zaprta posoda da se ogreje do sobne temperature, kjer se s Piranijevim vacuummetrom meri tlak, ki je enak prvotnemu parcialnemu tlaku vode v vzorcu. Metoda omogoča meritve parcialnih tlakov do nekaj tisočin tora v večini tehnično uporabljenih plinov.

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