

# **NATIONAL BUREAU OF STANDARDS REPORT**

10 609

Progress Report  
on  
**ADSORPTION OF WATER ON TOOTH  
COMPONENTS AND RELATED MATERIALS**



U.S. DEPARTMENT OF COMMERCE  
NATIONAL BUREAU OF STANDARDS

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## NBS PROJECT

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Progress Report  
on  
**ADSORPTION OF WATER ON TOOTH  
COMPONENTS AND RELATED MATERIALS**

By

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U.S. DEPARTMENT OF COMMERCE  
NATIONAL BUREAU OF STANDARDS



# ADSORPTION OF WATER ON TOOTH COMPONENTS AND RELATED MATERIALS

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## INTRODUCTION

The adsorption of water vapor especially in the study of biological systems has attracted considerable attention in recent years. Hagmassy, Brunauer, and Mikhail [1] have submitted that nitrogen is of only limited value in characterizing the surface properties of hardened portland cement pastes, silica gels, clay minerals, soils, etc., as compared with water vapor adsorption. They stated, ".....Not only are the smallest pores left out of consideration by nitrogen adsorption, but there is definite evidence that nitrogen cannot penetrate into many large pores....." This is consistent with some of the observations regarding the surface characteristics of dentin: The surface area, measured by the conventional nitrogen adsorption, gave rise to values of about ten square meters per gram. [2] However, upon extraction of the collagen, the anorganic dentin exhibited an area available to nitrogen of more than a ten-fold increase. [3] What is more significant, however, is that untreated dentin when measured by a polar molecule such as ammonia exhibited a surface area of  $138 \text{ m}^2/\text{g}$  which was more in agreement with the nitrogen value [4] for anorganic dentin.



If water vapor adsorption could be performed reliably, expeditiously, and with a sufficiently simple experimental apparatus, its use would be commonplace. This paper utilizes an extremely simple apparatus to study the adsorptive characteristics of water vapor by tooth components and related dental materials. It shows how surface areas obtained by this means compare with the results obtained using other adsorbates. Furthermore, it delineates the complete adsorption-desorption hysteresis loop for each material studied. In certain selected instances scanning<sup>[5]</sup> curves are also represented.

## EXPERIMENTAL \*

Both volumetric and gravimetric methods have been used in the past in adsorption studies. Gravimetric is preferred in water vapor adsorption because water is a liquid at room temperatures where the measurement of volume of vapor becomes unwieldy in any appreciable quantity because of its low vapor pressure. Gravimetric adsorption has the further advantage of being a direct measure of the amount adsorbed and not subject to cumulative errors which are ever present in the volumetric procedure where the incremental additional amount adsorbed at each point is added to that already there.

The course of the adsorption process can be followed continuously if the balance is an integral part of the adsorption apparatus and several different balance schemes have been used successfully in this connection. Commercial equipment is even available for those who prefer it. It is not the purpose of this paper to discuss the pro's and con's of the many methods which have been used over the years with various degrees of success. Nevertheless, their advantages and disadvantages were considered very carefully before deciding on the circulating carrier gas method used in this work.

The schematic outline shown in Table 1 (without making any claims as to its completeness) serves to illustrate the various methods reviewed .

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\* Certain commercial materials and equipment are identified in this paper to specify adequately the experimental procedure. In no instance does such identification imply recommendation or endorsement by the National Bureau of Standards or that the material or equipment identified is necessarily the best available for the purpose.

The present apparatus (Fig. 1.) uses air as the carrier gas. In its circulatory path, it passes from the pump through the saturator<sup>[14]</sup> containing one of more than a dozen different saturated salt solutions<sup>[15,16,17,18]</sup> which fixes the relative humidity. From here the gas-vapor mixture passes upward through the sample tube where equilibration takes place; then back to the pump to complete the cycle. A "Sigma Motor" pump with "tygon" tubing assures that the circulating vapor contacts no metal, valves, or foreign lubricants and its total volume is kept desirably small. Multiple experiments are simultaneously performed where not only the samples are independent, but the humidities are different, as well. The equipment, moreover, possesses a "fail-safe" attribute in the event of a power outage or pump malfunction. Once a day the sample tubes are capped at both ends with their respective ground glass joints and removed for weighing. Several days may be required in some instances for a single point, depending upon the nature of the sample and the closeness to saturation or dehydration on the humidity scale. An analytical balance or micro balance can be used depending upon the size of the sample. Both the "Gram-Atic" and "Micro Gram-Atic" balances (Mettler Instrument Co.) were used in this work.

Where the changes in weight observed from one relative humidity to the next were especially small, it would be necessary to make a correction for the difference in density between dry air and humid air which occupied the dead space of the sample tube. This correction  $\epsilon$  which was added to the apparent weight at each relative humidity may be determined from the relationship:



$$\epsilon = (0.014)(\text{R.H.})(V_d) \text{ milligrams.}$$

Here,  $V_d$  is the dead space volume (ml) of the sample tube and R.H. is the relative humidity expressed as a decimal.

The cross-sectional area of the water molecule used in this work was 12.5 square angstroms as given by McClellan and Harnsberger<sup>[19]</sup>. The BET "free surface" isotherm equation was used for determining surface area within the applicable range of humidities. Only the adsorption boundary curve of the hysteresis loop was used for these calculations<sup>[20]</sup>. Zero humidity was accomplished by substituting for the saturator a vertical cylindrical tube containing  $\text{P}_2\text{O}_5$  supported on glass wool. The lower end of the tube was fitted with a trap to catch the liquid phosphoric acid which resulted from the uptake of water. At least a week was required before a sample could be considered dry as evident by no additional loss of weight.

The order in which the points were obtained for an entire adsorption-desorption isotherm hysteresis loop generally consisted of desorbing from a 100-percent humidity environment in a stepwise manner until zero humidity was attained. Then adsorption was performed incrementally using each saturator, in turn, to obtain successively higher humidities until 100-percent humidity was again approached. If only the surface area were required, it would have been sufficient to take the sample to dryness initially before obtaining the adsorption points and then adsorbing only to the upper limit of the BET range of relative humidity. If more detail was desired such as the development of a scanning curve, this would be obtained on the second time around. The hysteresis loop would be retraced until at some point along the way the direction would be changed (either from desorption to adsorption or vice versa) and the new direction pursued in a stepwise fashion as before.

## RESULTS

Figures 2, 3, 4, 5, 6, and 7 show the complete adsorption-desorption hysteresis loops for collagen, dentin, enamel, anorganic whole teeth, and polymethyl methacrylate (denture base material), and investment compound, respectively. The adsorption isotherm which is the lower curve in each case is the basis for determination of surface area. It is over a portion of this curve that the familiar free-surface BET equation [21] is applicable. The weight of water adsorbed at relative humidity  $x$  is, of course, obtained by subtracting from the weight of the sample, after obtaining steady-state (and correcting for dead space if need be), the weight of the dehydrated sample. This is then divided by 18 times the dehydrated sample weight to convert it to  $N$ , the number of moles of water adsorbed per gram of dry sample. The isotherms (Fig's 2 through 7) were obtained by plotting  $N$  against  $x$ . The BET equation:

$$\frac{x}{N(1-x)} = \left[ \frac{(C-1)}{(N_m C)} \right] \cdot x + 1/(N_m C) \quad . . . (1)$$

limited to the range 0.08 to 0.33 in  $x$  was used to obtain the surface area available to the water molecule. Surface area is proportional to the parameter  $N_m$  which, in turn, is evaluated from the slope and intercept of the straight line which results from plotting the quantity  $x/[N(1-x)]$  against  $x$ . The reciprocal of the sum of the slope plus intercept gives  $N_m$  directly. The factor  $7.529 \times 10^4$  when multiplied by  $N_m$  (moles/g) results in surface area ( $m^2/g$ ) when the adsorbate is water vapor at room temperature as it is in this work.

The surface areas obtained by following these steps are shown in the top row of Table 2 compared with results for other adsorbates on the same materials. <sup>[4]</sup> It is significant that large increases were found for surface areas available to the water molecule as compared with the nitrogen molecule for dentin as was found for collagen. Even enamel showed a ratio of about two to one in this regard. In each case where the discrepancy is large it can be attributed to or protein-type material the presence of collagen as one of the components. In contrast, anorganic whole teeth showed little change from one adsorbate to another.



## DISCUSSION

When a greater amount of adsorbate is observed during desorption at the same relative pressure as was found during adsorption at constant temperature, the phenomenon is termed "adsorption-desorption hysteresis". It is observed with other adsorbates besides water, although water hysteresis is frequently quite pronounced. Even with water in some instances hysteresis may be absent. Its causes are only partially understood. A different mechanism may very well be controlling at low relative pressures than at intermediate or high values<sup>[26]</sup>. In the latter case, capillary condensation within the pore structure is the reason most frequently cited by investigators<sup>[27]</sup>. The myriad of shapes and sizes of pore openings could easily account for many types of hysteresis loops. Everett<sup>[28]</sup> classified four types of hysteresis independent of the shape of the isotherm. Type A shows the loop confined to the mid-portion of the relative pressure range. In type B the loop extends all the way to saturation, but is absent in the lower range of pressures. Type C has the loop extending over the entire range of pressures; while type D appears to be intermediate between types B and C.

Relating to the present investigation, it appears that denture base material, Fig. (6), might represent type A or B. An interesting aspect of this adsorbent is that the adsorption isotherm, itself, has no inflection point and is always convex to the pressure axis. This is characteristic of type 3 isotherms (Brunauer's classification)<sup>[29]</sup> in which the heat of adsorption is less than the heat of liquifaction of water. In other words, the methyl methacry-



late polymer would appear to be hydrophobic.

Enamel, Fig. (4) , would probably qualify as a type B hysteresis. The same would seem to be true for anorganic whole teeth, Fig (5), although the choice between types B and D is probably a matter of degree.

*Fig (7)*

The Durallium investment compound<sub>A</sub> is an interesting case that deserves special comment. The initial desorption from saturation produced a curve which fell below the subsequent adsorption curve originating from zero humidity! A second desorption, however, using the same sample resulted in a higher curve as it should have been. The initial abnormal behavior indicated some irreversible change taking place in the adsorbent. Its history<sup>[30]</sup> disclosed that it possessed a vitreous (or glass-like) phase as a result of its high-temperature preparation. It was therefore quite possible that over the period of weeks required for a complete desorption-adsorption cycle, the condensed phase of adsorbed water had actually dissolved some of the vitreous material. Upon continuing the desorption, the water vapor which was removed left behind salts as a precipitate, together with irregularities in the surface of the glass-like phase. Both of these processes would have resulted in increasing the surface area of the sample which would manifest itself by disclosing a higher adsorption curve. This phenomenon of more than doubling the surface area of newly formed glass as a result of water washing has been known for many years<sup>[6]</sup>. Once reacted with water the sample, of course, became stabilized.

The hysteresis loops for rat-tail collagen and for dentin, Figures (2) and (3), are excellent examples of type C. It is an

interesting observation that swelling is also known to occur with these two systems during water adsorption. Other systems which have been reported where swelling occurs such as ammonia on silk fibroin<sup>[31]</sup>; water on casein<sup>[32]</sup>; water on gelatine<sup>[33]</sup>; water on collagen<sup>[2,34]</sup>; and polar gases on certain clays<sup>[35,36]</sup> (to mention a few) all share in common the fact that they exhibit type C hysteresis. It is significant that when the collagen is removed from a tooth, the type of adsorption hystereses obtained with water vapor changes from type C to B. Correspondingly, appreciable dimensional changes as a function of relative humidity are also drastically reduced virtually to zero as found by [Dickson and Barton]<sup>[37]</sup>. Other materials which have been found to exhibit dimensional changes with humidity<sup>[38,39,40]</sup> have demonstrated the existence of dimensional hysteresis, as well. There is every reason to believe that dentin would behave in a similar manner. If so, the method of drying a tooth prior to the application of an adhesive or direct filling resin, etc., would merit critical reexamination.

This discussion of adsorption hysteresis has so far been confined to the boundary curves which comprised the outer envelope of the isotherm plot. It is possible by means of an infinite number of different paths to reach any point within the loop. Such paths are commonly referred to as scanning curves<sup>[5]</sup>. They are obtained by changing direction (adsorption to desorption or vice-versa) before reaching either end of the isotherm. One such scanning curve is shown for collagen, Fig. (2), where the process was changed from desorption to adsorption before the sample was completely dried. It occurred at a relative humidity of 0.04. The scanning curve

resulting from continuing the adsorption beyond this point remained within the loop distinct from either boundary curve until they converged at (or near) saturation. This is a characteristic behavior shared by all primary scanning curves. [40]

Where the hysteresis loop extends throughout the entire range of the adsorption isotherm and is sufficiently pronounced as is the case with dentin, an excellent opportunity is afforded to explore the behavior of scanning curves in detail. This aspect of the investigation has evolved to such an extent that it deserves to be treated as an entity in its own right.

One important aspect of the hysteresis studies, however, was the fact that the hysteresis loops could be retraced on subsequent cycles. There were no indications of degeneration as have been reported by others [40]. Good reproducibility was also observed from sample to sample for the same material.



## CONCLUSIONS

An apparatus, simple to construct and based on a recirculating system, has been used with multiple samples for obtaining water vapor adsorption isotherms gravimetrically. While having ruggedness as well as fail-safe properties, it possesses accuracy and reproducibility.

The complete adsorption-desorption isotherms were obtained for collagen, dentin, anorganic whole teeth, polymethyl methacrylate, and a commercial investment compound.

Surface areas available to the water molecule were determined from BET plots based on data obtained from the appropriate portion of the respective adsorption boundary curve. Complete drying with  $P_2O_5$  was a prerequisite for reliable surface area evaluation. Collagen and collagen-containing materials showed the greatest increases in surface area available to the water molecule compared with nitrogen. These areas (measured by water vapor adsorption) were in reasonably good agreement with previously obtained values on the same samples using  $NH_3$  as the adsorbate.

Well defined and reproducible hysteresis loops were found and characterized according to type. The presence of collagen in appreciable quantities in teeth appeared to be responsible for the persistence of the hysteresis loop down to the lowest humidities. No degeneration of the hysteresis loops was found for those cases (collagen and dentin) where the experiment was repeated. The anomalous initial hysteresis behavior of investment compound could satisfactorily be explained based on an interaction with adsorbed water. Primary scanning curves were obtained in selected instances and (after initiation) remained distinct from either boundary curve.



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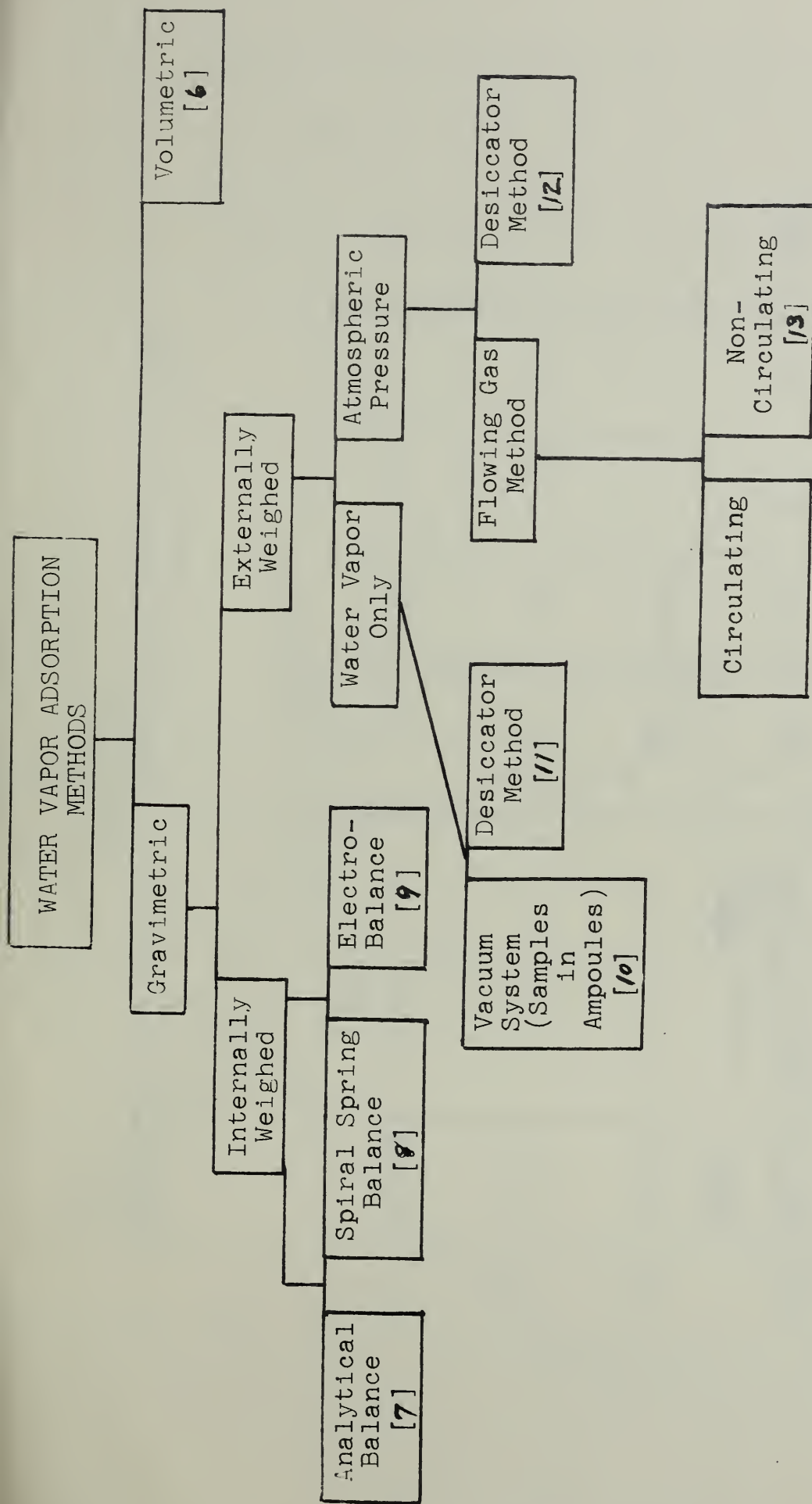


Table 1 Classification of Water Vapor Adsorption Methods

	Collagen (Hide) (m <sup>2</sup> /g)	Collagen (Rat tail tendon) (m <sup>2</sup> /g)	Dentin (m <sup>2</sup> /g)	Anorganic/ whole teeth (m <sup>2</sup> /g)	Enamel/PMMA (m <sup>2</sup> /g)	Invest- ment Comp'd (m <sup>2</sup> /g)
H <sub>2</sub> O (23 C)	408 [22] 470 [23]	425 450 454	128	75	8.7	30 0.86
NH <sub>3</sub> (-33.4)	- - -	- -	138	67	- -	- -
CO <sub>2</sub> (-78.4)	- - -	- -	50	48	- -	- -
N <sub>2</sub> (-195.8)	< 5 [24] 1 [25]	- -	7.5	64	4.0	- -

Table 2 BET Areas (based on 12.5 square anstroms for the adsorbed water molecule) Compared with Results for Other Adsorbates.

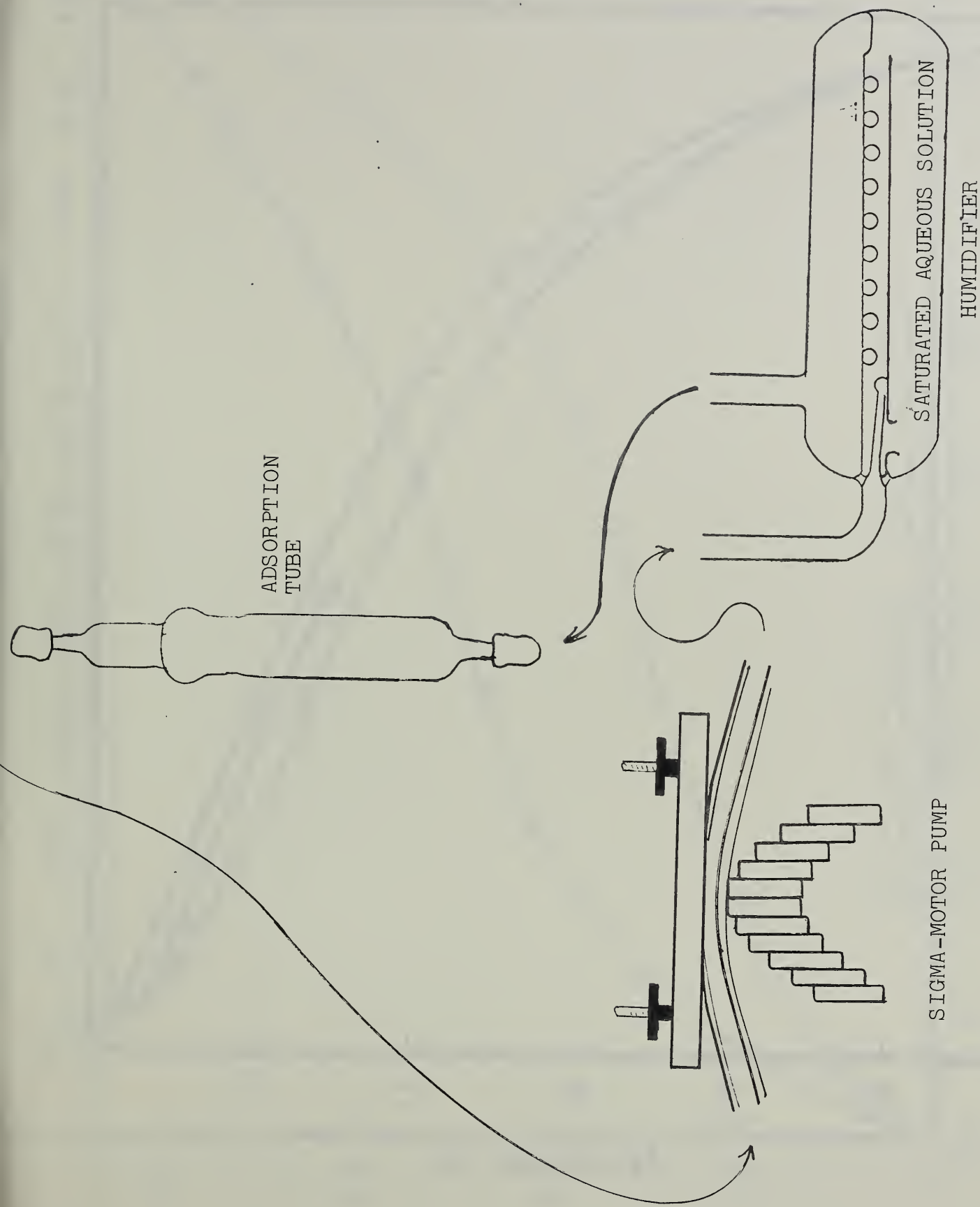


Figure 1. Circulatory water vapor sorption apparatus.

Figure 2. Sorption isotherms for water on collagen at 23°C. Light circles are adsorption points--dark circles, desorption. Complete hysteresis loop is shown along with a primary scanning curve.

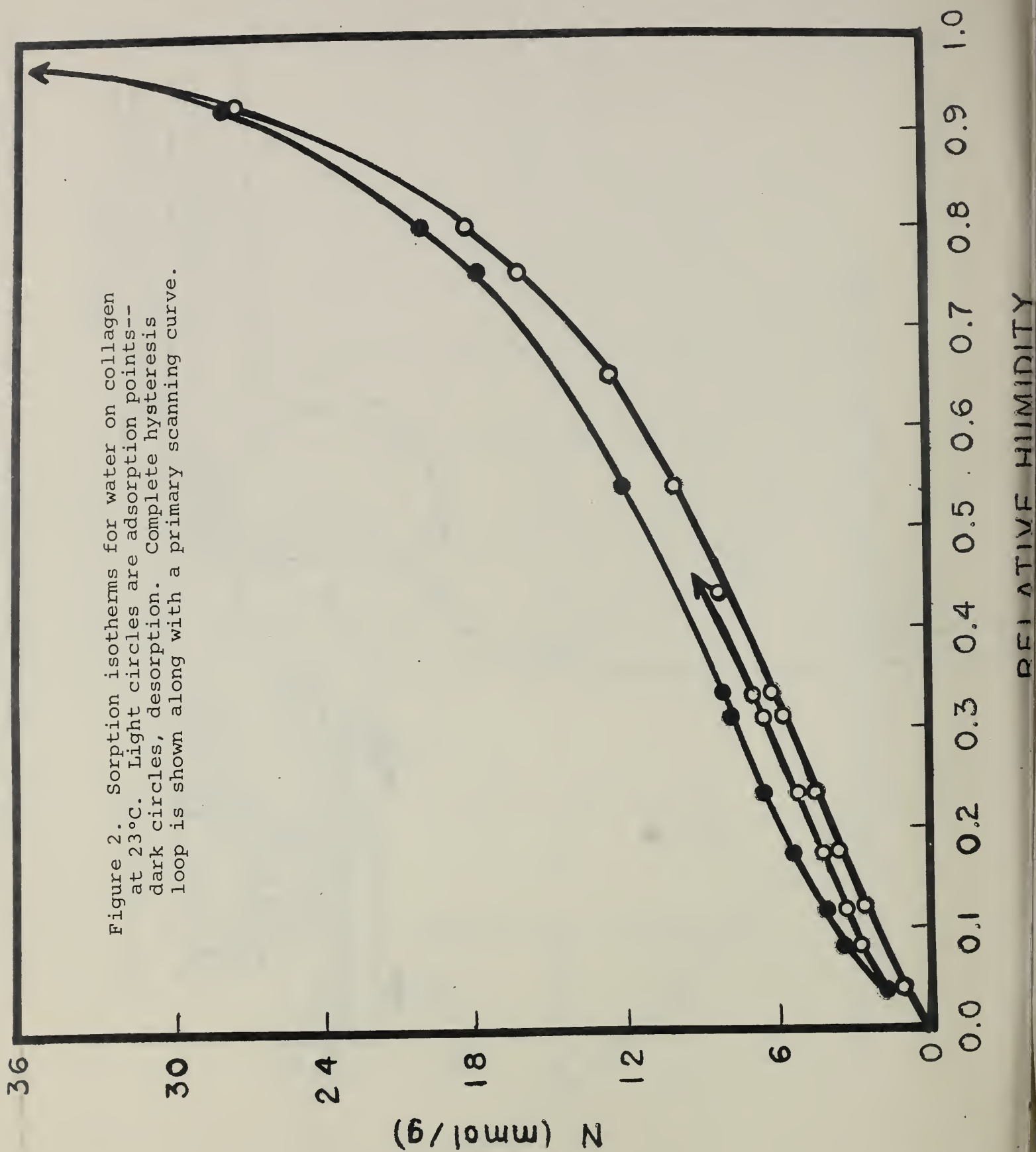




Figure 3. Sorption isotherms for water on human dentin at 23°C. Light circles are adsorption points-- dark circles, desorption.

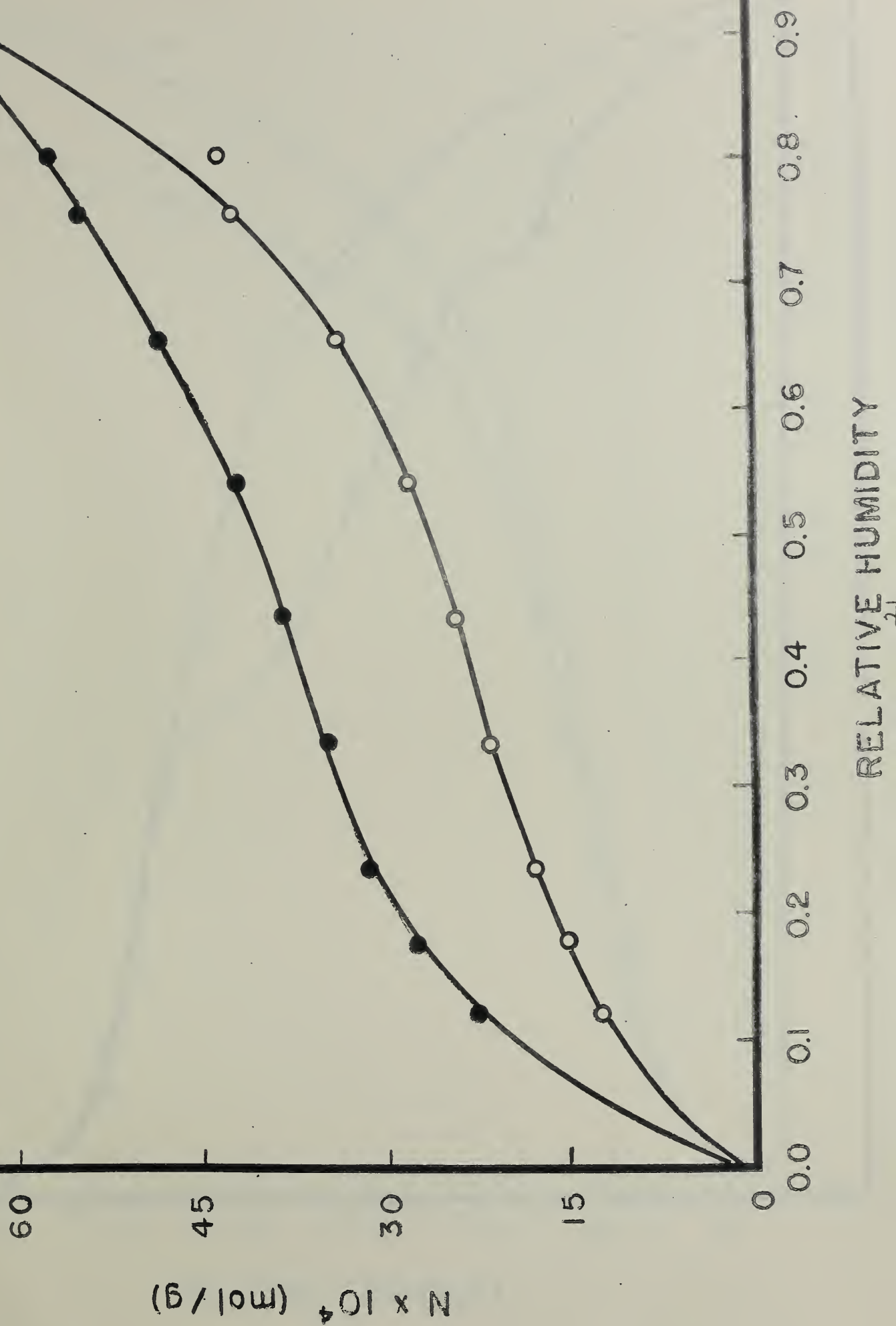


Figure 4. Sorption isotherms for water on human enamel at 23°C. Light circles are adsorption points-- dark circles, desorption.

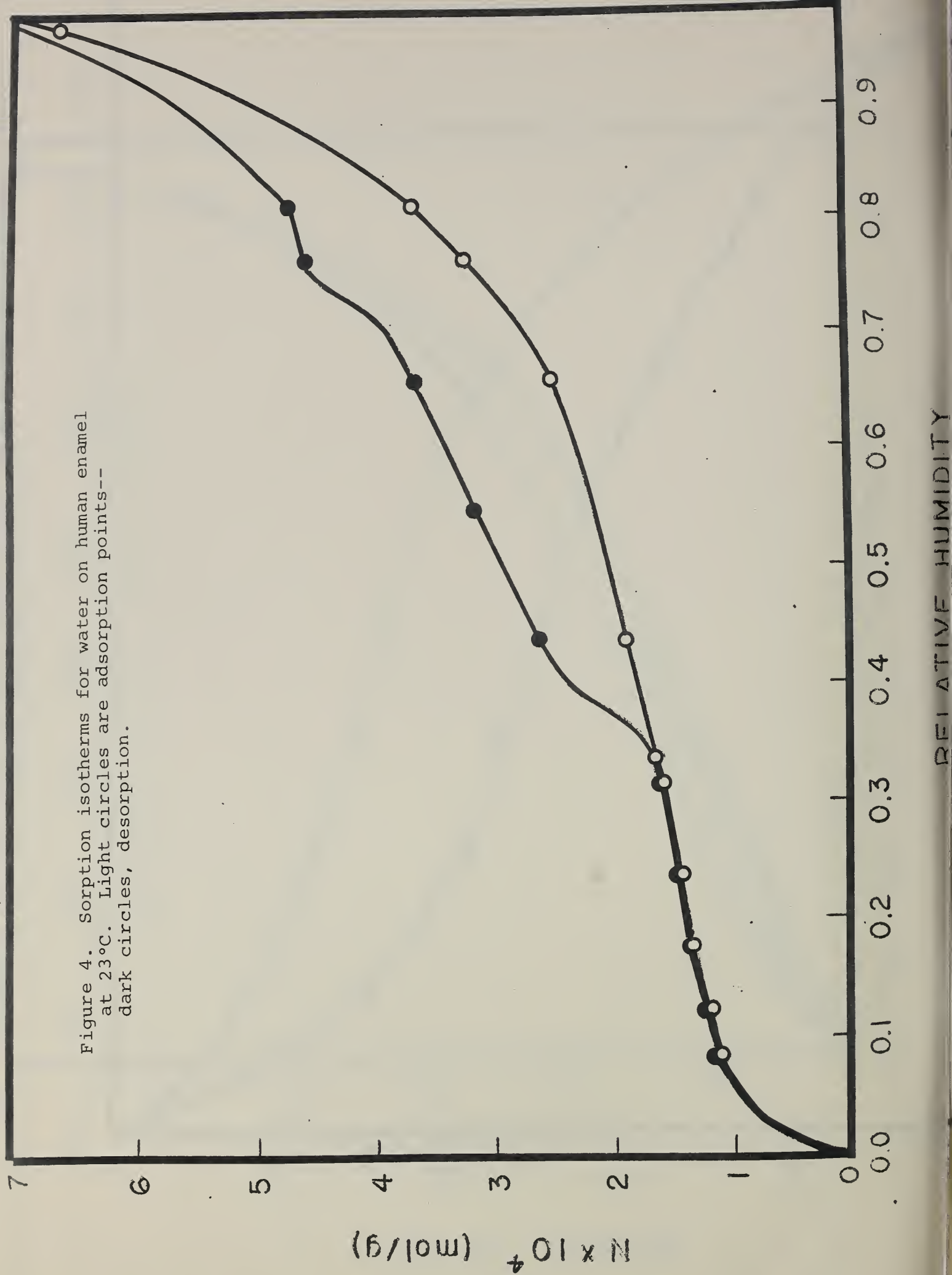


Figure 5. Sorption isotherms for water on anorganic whole teeth at 23°C. Light circles are adsorption points--dark circles, desorption.

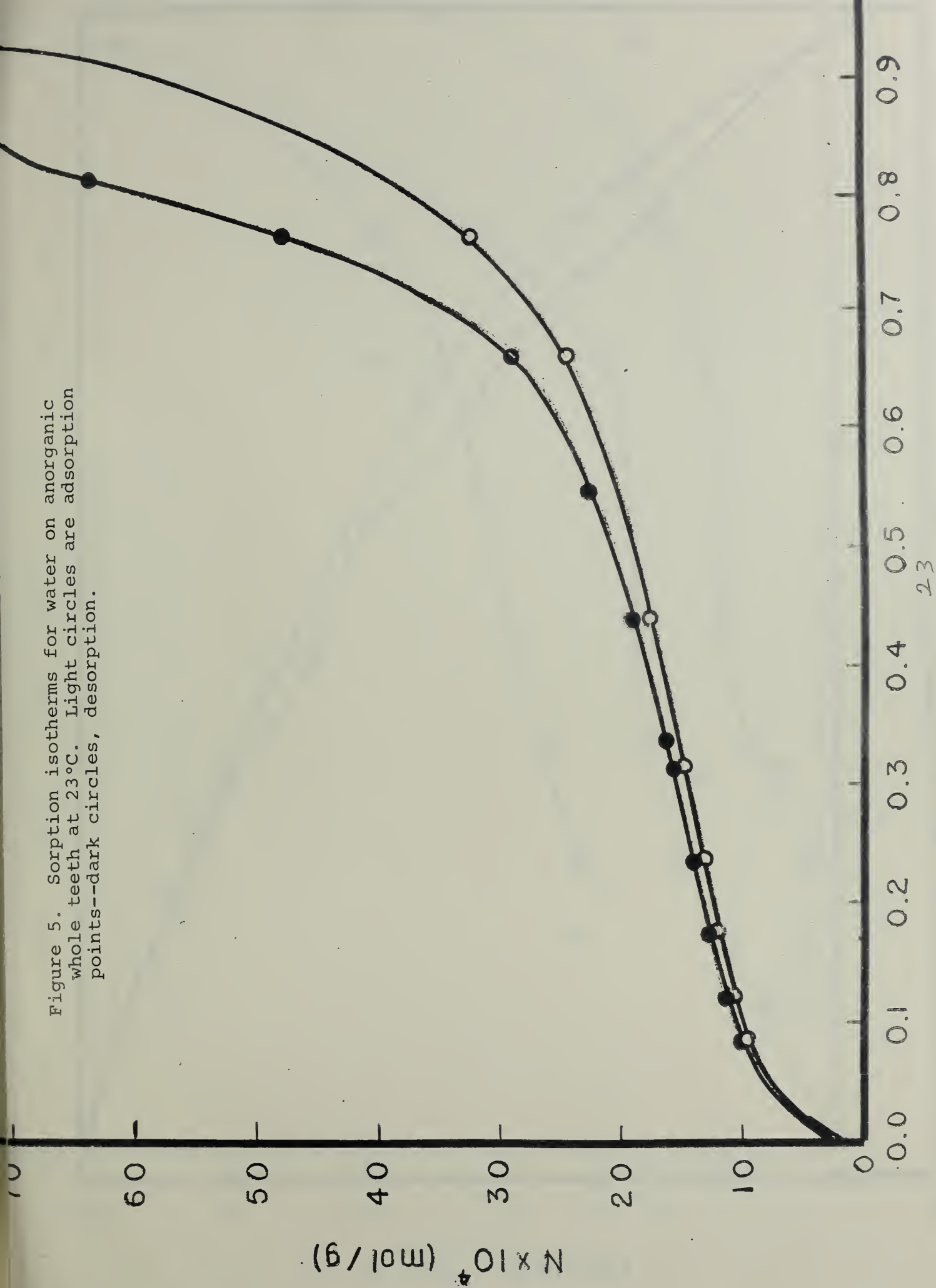


Figure 6. Sorption isotherms for water on polymethyl methacrylate (denture base material) at 23°C. Light circles are adsorption points -- dark circles, desorption.

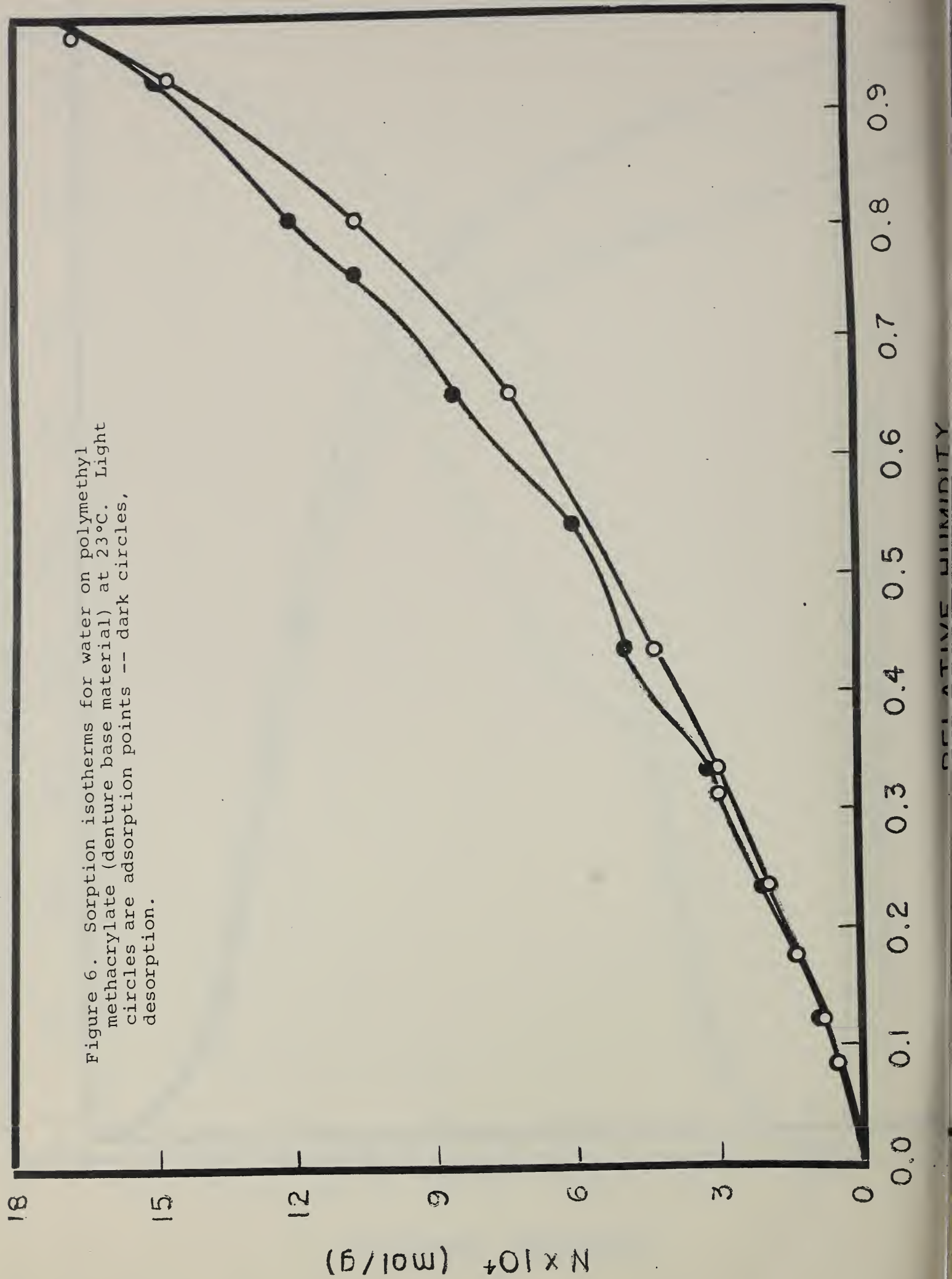




Figure 7. Sorption isotherms for water on a high-temperature fired commercial investment compound at 23°C. Dark circles are the initial desorption points; light circles show the subsequent adsorption points (after dehydration); dark triangles depict the final desorption points.

