PECULIAR PROPERTIES OF WATER 
AND THE CRYSTALLINE STRUCTURE OF ICE

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ANOMALIES OF WATER AND THE CRYSTALLINE STRUCTURE OF ICE

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V. A. Al'tberg

Water, as is well known, exhibits an entire order of highly remarkable physical anomalies which have an important significance for the physical life of the land. These peculiarities and anomalies, placing water in a special position among other substances, are found, as will be seen below, closely related to its molecular structure. They depend on, first of all, the variations in this structure in relation to the external physical conditions (temperature and pressure) and, secondly, the nature of its chemical components (H, OH). This report gives a brief summary of new data concerning the structure of water and related anomalous properties, and also the structure of crystalline ice and its several types.

Of the physical factors affecting the properties of water, we shall first consider temperature. Almost all of the peculiarities of the physical properties of water are connected with its thermal condition and usually appear at lower temperatures (below 0°C), whereas at higher temperatures water possesses normal properties. Such a change in the properties of water, as will be seen later, is directly related to the changes in its molecular composition, and with the formation during cooling of doubled, tripled, and more complex molecules of the type \( \text{H}_2\text{O}_n \).

Of these peculiar properties, attention was long ago directed towards, first of all, the anomalous relationship between the density of water and temperature near the melting point. It is
well known that water, as distinguished from all other liquids, when heated from 0°C to 4°C does not expand but contracts, and only after further heating above this temperature does it expand like other liquids. The reason for such a variation in water density, having a great significance in nature (it causes, among other things, rivers and lakes to freeze from the top, and not from the bottom), was long ago sought by attempting to find in the formation of water during cooling special kinds of ice-forming molecules of lesser density.

Roentgen was the first to develop at great length the concept of two kinds of molecules in water. According to this concept, during cooling of water there develop special molecules having the properties of ice and, therefore, less density, and their number increases as the water temperature becomes lower. As a consequence, the increase in quantity of these special molecules of lower density appears as a decrease in the overall density of the water. As water is cooled, two processes take place simultaneously: the usual contraction with decreasing temperature and a process in opposition to it, an expansion due to the increase in the number of molecules of lower density. The first of these processes prevails down to 4°C, and the second below 4°C. On the boundary of these two temperature regions, therefore, water density is at a maximum. At lower temperatures, according to Roentgen, water may be considered as a solution of ice in water. This assumption also accounts for the effect of pressure on the temperature of maximum density, on the freezing point, and on water viscosity. Roentgen considered it useful to adopt the same
view, i.e., two kinds of molecules, in relation to gaseous and solid substances. Such a proposition was later confirmed, at least in relation to substances tending towards polymerization not only in the liquid state, but also in gaseous and solid states. The concept of the formation of complex molecules was later developed by von Laar, Sutherland, Duclaux, and, in particular, Tammann, whose opinions and recent work will be discussed later.

The interrelationships among molecular weight, composition, and molecular structure and the physical properties of gaseous substances have been well-defined in the kinetic theory of gases. Less is known, however, about liquids. Whereas the behavior of single substances in gaseous and liquid states is rather well-described by the well-known theory of Van der Waals, coming from the idea of continuity of both states and the assumed invariability of the molecular structure during transition from one state to another, the behavior of other substances, with their transition into a liquid state, does not agree at all with this theory.

Their behavior does not agree, apparently because the premise about the invariability of the molecular structure for these substances is not applicable, inasmuch as liquid molecules during transition become complex and associated. From this point of view, liquids may be divided into two large classes: normal, having identical molecules in both liquid and gaseous states, and anomalous,
or associated liquids, the molecules of which in the liquid state are complex and represent groups of simple molecules. A good example of the second class of liquids is water.

In order to solve the question of the degree of molecular complexity, it is necessary to examine the interrelationships between molecular weight and physical properties of liquids. One of these interrelationships between surface tension (in dynes), molecular volume, and temperature was empirically established by Eötvös as early as 1886. In the somewhat altered form of Ramsay and Shields, the relationship has the following simple form:

\[ \gamma \nu^3 = k(T-d) \]  \hspace{1cm} (equ. 1)

where \( T \) is the difference between observed and critical temperatures, \( d \), a number close to 6, and \( k \), the temperature coefficient of molecular energy of the surface. The comprehensive investigations of Ramsay and Shields confirm the correctness of the relationship noted by Eötvös and demonstrate that the coefficient \( k \) for the majority of liquids is constant and equal to 2.12; for water, alcohol, and acid, on the other hand, this coefficient is less (for water, as an example, about 1). For satisfying the formula given, as calculations show, one must allow multiple molecular weights, as, for example, \((H_2O)_3\), \((CH_3OH)_3\), etc.

In this way only an average molecular weight is calculated; in reality, associated liquids may exhibit a mixture of molecules with various molecular weights, so that the relative quantities of these and other molecules change rather easily with temperature. If \( n \) signifies the factor of association, showing how
many times the molecular weight of the given liquid is greater than the molecular weight of a corresponding usual formula, then for water the dependence of this factor on temperature is given in Table 1:

(Table 1)

In view of the change in molecular composition during changes in the aggregate state, associated liquids, especially water, do not fit at all into the framework of Van der Waals' theory and give cause for an entire order of deviations from the rules and conformity to principles established for normal liquids (the rule of Cailletet and Matias, the rule of Trauton, the conformity to principle concerning the critical density, which for water exceeds by 3 to 4 times the magnitude expected theoretically).

A parallel is observed between the association of molecules and the dielectric constant of liquids: as the association of molecules becomes greater, the given liquid has a greater dielectric constant. In addition to the dielectric constant, a favorable factor for the association of molecules is the increase of internal liquid pressure. Both of these factors, instrumental for the association of molecules, appear especially strongly in the case of water, which has a maximal dielectric constant (80) and colossal internal pressure, measuring tens of thousands of atmospheres.

The foregoing indicates the strong tendency of water towards association of its molecules, whose complex character is also indicated by its highly complex absorption spectrum, consisting of multiple bands which characterize the inherent oscillations of
various parts of a complex molecule. Still another anomaly of water (see below) is anomalous dispersion, which is especially marked in the case of water.

Of other substances which illustrate well the strong association of atoms, sulfur may be cited, vapors of which at a temperature of 1000°C are 2-atom, at lower temperatures 4-atom, and near the point of condensation even 8-atom.

Other characteristic properties of water to be noted are its strong dissociating (thanks to the significant dielectric constant of water) and dissolving abilities and also its unique catalytic effect in many chemical reactions, so that water is in a sense a universal catalyst, playing a large role in nature.2


Moreover, water is also sharply distinguished from all other liquids by a very high mobility of its free ions:

\[ H = 315, \text{ and } OH = 174. \] (equ.2)

For other liquids the movements of ions compose in all several tens of the same units.

Much new data on the structure of water in a solid state has been introduced by the investigations of Tammann and Bridgman. At first Tammann discovered two new types of ice, which he called Types II and III (Type I is ordinary ice). Compressing ordinary ice at -30°C, he discovered that at a pressure of 2500 kg/cm² the pressure quickly falls to 2200, during which the volume of water
decreases by almost 20%, after which the pressure remains const-
stant for some time. During such a decrease in volume, ordinary
ice becomes more dense, Type III, which is denser than water.
Submerged in liquid air, it sank, whereas Type I floated in liq-
uid air. Tammann once obtained what seemed to be yet another
version - Type IV. Its existence, however, remains doubtful, in-
asmuch as no one, not even Tammann himself, was able to create
this type a second time.

Tammann conducted these experiments at pressures up to
3000 atmospheres. Bridgman went much further, namely to 20,670
atmospheres and discovered two more new kinds of ice, Types V and
VI. Curves of the melting of various kinds of ice, according to
Bridgman's data, are given in Fig. 1.

Fig.1. Curves of the melting of various types of ice.
Key: 1-kg/cm².

Of all types of ice, only Type I, as opposed to all other
substances, has a melting point which decreases with increasing
pressure; for all other types, the melting point increases nor-
mally, reaching 76.35°C at a pressure of 20,670 kg/cm². At such
a high pressure genuine hot ice is formed.

The conclusions of Bridgman concerning the theory of liquid
and solid states are interesting. There are, as is well known, two
such theories, of which the first (Planck, Poynting, Ostwald,
and others) considers that continuous transformation of the liquid
phase into the solid is possible when the process takes place
above the critical point. The second theory (Damien, Tammann)
does not recognize, in general, the existence of critical points for liquid and solid states. Liquid and gas, according to Tammann, are distinguished only quantitatively, by the distance between molecules. The solid state is distinguished from the first two states by the oriented distribution of molecules in space, as in a crystalline lattice.

Instead of the theory of critical points, Tammann proposes the theory of maxima for closed melting curves. The investigations of Bridgman, however, show that the curves of melting do not show the existence of maxima, despite the fact that the region of applied pressure significantly exceeded those limiting pressures at which the curves of melting should have, according to the theory, passed over the maximum. In fact, the investigatory curves of Bridgman go to infinity according to an unknown law. Neither of the two theories referred to found confirmation in the important investigations of Bridgman, who, therefore, summarily rejected both theories. His idea, founded on his experiments, led to the fact that at high pressures, molecules of a liquid can assume a definite orientation, so that some molecules have an oriented structure, while the other molecules exhibit random and disorganized motion. As pressure is increased, those parts of the water with oriented atoms grow in number and size, continually absorbing more and more atoms until all water atoms become oriented, i.e., until all of the water changes into the solid state. Indeed, Bridgman was able with an increase in pressure alone without simultaneous cooling of the water to transform it into the solid state at temperatures sig-
significantly higher than the freezing point. Thus, at a pressure of 20,000 atmospheres even hot water at a temperature of +76°C changes into ice, not ice. Also, liquid oils can be transformed into the solid state by high pressure alone.

In connection with the question as to what molecules transform the above-mentioned types of ice (I, II, III, V, and VI), Tammann categorically states that the formation of each type depends on a special kind of molecule corresponding to that type which is present in the liquid long before crystallization. According to this opinion, water is a mixture of several kinds of molecules, each of which under appropriate conditions produces a corresponding type of ice.

The opinions of Tammann on the structure of water are stated in more detail in one of his recent papers which we will now consider in greater detail. According to Tammann, ordinary ice develops from Type I molecules which are more numerous than all other types. In water they are formed long before crystallization, but their quantity increase is especially great near the solidification point of the given type of ice. Their presence explains the anomalous behavior of the density of water between 0°C and 4°C. With the discovery of special types of ice having a greater density than water, this opinion, expressed long ago, received new confirmation. As water is subjected to increasing pressure, the quantity of Type I molecules decreases and crystallization is not in the form of ordinary Type I ice, but in the
form of Type III ice. This type is denser than water and consists of Type III molecules which are more dense than Type I.

The presence in water of Type I molecules explains the anomalous character of the dependence of all properties on pressure and temperature, so that the concentration of Type I molecules may be expressed as a function of pressure and temperature. Calculations show that at a temperature of 0°C and pressure p=1 atmosphere about 1/5 of the water consists of molecules of this type. At 50°C their concentration is much less.

Molecules in water other than Type I are much more difficult to identify and distinguish.

Tammann determined the concentration of Type I molecules from curves of the relationship between the specific volume and temperature of water at constant pressure p=1 (Fig. 2).

<table>
<thead>
<tr>
<th>Key</th>
<th>1-a</th>
<th>2-b</th>
<th>3-c</th>
<th>4-d</th>
</tr>
</thead>
</table>

Fig. 2. Specific volume of water vs. temperature.

In the temperature region between 100°C and 70°C this relationship is almost a straight line, with a very low constant curvature (0.006 in 10°C). Beginning with 70°C the curvature becomes greater with lower temperatures due to an increasing concentration of Type I molecules. If, beginning at 70°C, the upper portion of the curve is continued, it will retain its earlier low curvature (0.006 in 10°C), and the quantity of water (without Type I molecules) at 0°C is 0.089 cm³.

Considering that the quantity of ice at the same temperature
is 1.090 cm³. upon transformation of water into ice there is a volumetric increase of 0.090 cm³.

Proceeding from these data, Tammann found the following distribution of concentration C of Type I molecules at various temperatures in one gram of water:

(Table 2)

At \( t = 0°; 10°; 20°; 30°; 40° \)

Concentration C = 0.15; 0.12; 0.08; 0.06; 0.04 in one gram of water

The concentration of the remaining four types of molecules, from which the above-mentioned four stable kinds of ice are formed, also depends on \( n \) and \( t \). Near the melting point of each of these types of ice, water is relatively rich in that type from which a corresponding type of ice will be formed. All five types of molecules exist in mutual equilibrium, and during changes of \( t \) and \( n \) the conditions of equilibrium are rapidly displaced.

Later Tammann determined the degree of polymerization of Type I molecules and found their chemical formula to be \((\text{H}_2\text{O})_6\). The division of these enlarged molecules into 2 parts -- \((\text{H}_2\text{O})_3 + (\text{H}_2\text{O})_3\) -- gives that kind of molecule from which, for the most part, water is formed. The presence of Type I molecules appears not only as an anomalous relationship between the specific volume and temperature of water, but also in the unique effect of temperature on other such physical properties as specific heat, viscosity, surface tension, and others. We will now turn to the temperature changes of several of these properties.

Viscosity of water. Figure 3 shows, at three different temperatures \((0°C, 20°C, \text{and } 40°C)\), three curves indicating the
effect of pressure on viscosity of water (circles) and on friction, due to ion movement in a 0.1 normal solution (crosses). Both kinds of curves are congruent within the limits of observational error in the interval from \( p = 1 \) to \( p = 700 \text{ kg/cm}^2 \). Minima, occurring in all three isotherms, are explained as the consequence of a decrease in the concentration of Type I molecules according to the degree of pressure increase. Between \( 0^\circ \)C and \( 40^\circ \)C the relationship is linear at pressures above 2200 \text{ kg/cm}^2, in accordance with the condition that at high pressures water has very few Type I molecules. It also follows that at pressures above 2400 \text{ kg/cm}^2 only Type III ice, and not Type I, forms in water. Inasmuch as the concentration of Type I molecules decreases with \( t \), the minimum relative viscosity becomes less in slope and is displaced to lower pressures. With the disappearance of Type I molecules between \( 50^\circ \)C and \( 60^\circ \)C, as indicated by the volumetric isobar of water, there also disappears the minimum on the viscosity isotherm, which begins at once to increase without an initial decrease.

The effect of Type I molecules on viscosity may be demonstrated as follows: the straight sections of the isotherms are extended to intersect with the ordinates. The differences between the isotherms and the lines thus drawn indicate the effect of Type I molecules on viscosity.

A general analysis of the physical properties of water shows that their temperature relationships at high temperatures are simple and normal. Deviations for all properties of water begin at medial temperatures and increase with lower temperatures. A more satisfactory explanation of these anomalies is included in
the assumption that in water, as already noted, there is formed in great quantity a special kind of molecule. The temperature region of its existence lies between 50°C and the melting point of ice, and the pressure range within which these molecules exist is from 0 to 2500 kg/cm². Type I ice is formed from these molecules, whose formula is \((H_2O)_6\). The dissociation of such a molecule into two parts forms those molecules that are the main constituent of water and have the formula \((H_2O)_3\).

If experimental data pertaining to the structure of liquid water has no straight lines, then the presence of several kinds of molecules is indicated. In such a situation it is necessary to draw conclusions on the basis of indirect data. The study of water in the solid state and its crystalline structure is now considerably facilitated, inasmuch as the X-ray method from the time of Laue's discovery has provided us with a powerful instrument for determining the structure of substances. During the study of the structure of solid substances the above-mentioned method gave the most brilliant results, and promises for the future even greater usage and a more refined technique for study. In recent times this method has also been applied to the study of the structure of liquids, and in the near future will be applied to the study of gases and vapors.

For the study of the structure of ice the X-ray method was first used by John, then by Denison. The former found in ice a space lattice composed of orthogonal prisms of height \(h=6.65\) angstrom units (AU), with equilateral triangles in the base, a side of which is
\[ a = 4.74 \text{ AU.} \quad \text{(equ. 3)} \]

Ice for the experiments of Denison was placed into a Dewar flask with liquid air by means of a submerged glass tube, holding a small quantity of distilled water. Ice in the form of small crystals was exposed to X-rays for ten hours. Denison found the lattice structure to be similar to that determined by John, with the following prism dimensions:

\[ h = 7.32 \text{ AU, and } a = 4.52 \text{ AU.} \quad \text{(equ. 4)} \]

Neither of these investigations, however, determined the actual distribution of atoms in the lattice. This was done by Bregg with X-rays independently of a direct analysis. Bregg started with the assumption that ice belongs to that class of crystals in which the molecules consist of positive and negative ions, and that the structural arrangement is such that each positive ion is symmetrically surrounded by negative ions, and vice versa. In this way, the number of adjacent molecules is less, since it is well known that ice has a low density. Such a wide compass of space with a minimum of closely-contiguous molecules is possible if the oxygen atom is placed in the center of gravity of four other such atoms, arranged in the vertices of a tetrahedron, similar to the arrangement of carbon atoms in a diamond lattice. One hydrogen atom is located in each interstice, thus dividing two neighboring oxygen atoms. Such a structure requires twice as many hydrogen atoms as are required by the chemical formula of a water molecule.

The bonding of atoms in the crystal occurs in such a way
that the hydrogen ions give up their valence electrons to the oxygen ions. If the force holding atoms in the crystal is viewed in this way, the concept of a molecule loses its significance: the entire crystal may be considered a single huge molecule.

The dimensions of the lattice elements of ice can be determined by means of comparison with the analogous structure of the lattice elements of diamond. Oxygen atoms are substituted for carbon atoms and all hydrogen atoms are removed; although the molecular weight of a lattice element of ice is greater than that of diamond in the ratio of 18:12, the density of ice is less than the density of diamond in the ratio of 0.9165:3.52. Therefore, the linear dimensions of the ice lattice should be greater than those of diamond by the ratio 1:p.

For volumetric expansion,

$$p^3 = \frac{18\times3.52}{12\times0.9165}$$  (equ.5)

so that the linear expansion $p=1.79$.

The distance between centers of the two oxygen atoms in diamond is 1.54. Multiplying this by the coefficient of expansion $p$ found above, we get for the distance between centers of the two oxygen atoms $1.54\times1.79 = 2.76$. The distance between two adjacent basal planes in diamond is 2.05; the corresponding distance for ice is $2.05 \times 1.79 = 3.67$.

Finally, the distance between the two atoms located in one of the
above-mentioned planes is:

for diamond 2.52,

for ice \(2.52 \times 1.79 = 4.52\).

Dimensions of the ice lattice obtained independently by the X-ray method agree in accuracy with the dimensions obtained by Denison with the X-ray method, if it is taken into account that the height of Denison's prism is equal to twice the distance between two adjacent planes (III).

In order to clearly visualize the distribution of atoms in the ice space lattice, we shall use a flat diagram and model to represent the spatial structure of this hexagonal crystal. Figure 4 shows the arrangement of oxygen (white) and hydrogen

**Fig. 4. Schematic arrangement of oxygen (white circles) and hydrogen (black circles) atoms in an ice crystal.**

black) atoms in a region cut parallel to the base of the crystal. The black spheres are located in the plane of the diagram, the larger white spheres are in front, and the smaller white spheres are in back, behind the plane of the diagram.

In order to construct a complete model it is necessary to prepare several such structures, placing them parallel to each other so that lower white spheres of the upper structure face the upper white spheres of the lower structure. The black spheres are placed between the aforementioned spheres. Having connected the parallel structures with the black spheres, an entire model of an ice crystal can thus be visualized. It would be possible to construct the model in another fashion, by assembling it from
the simple prisms of Denison and Bregg (Figs. 5a and 5b) into the
form of a single cell, cut from the model without the spheres.

Fig. 5. Schematic arrangement of oxygen (white circles) and hy-
drogen (black circles) atoms in an ice crystal.

Having assembled the spheres, we now have an arrangement of
atoms in a simple prism of a lattice with absolute spacing between
the centers of the atoms (Fig. 5b), as determined by X-ray (John and
Denison) and by comparison with the elements of a diamond lattice
(Bregg). Such a model was prepared under our direction in the
Department of Experimental Geophysics of the Main Geophysical Ob-
servatory and is shown in the photograph (Fig. 6).

Fig. 6. Model of an ice crystal.

The model shows that the molecule is characterized by a
non-compact structure - the model has many vacant spaces unoccu-
pied by molecules. This explains the low density of ice (less
than that of water), and it graphically demonstrates the results
of Bregg's above-mentioned calculations.

Thus, the X-ray method provided consistent and accurate de-
terminations of the absolute distances between atoms, their posi-
tions in crystals in general and in ice crystals in particular.
A further step in this direction was taken by Debye and Sherer,
who used this method to study the structure of powder crystals,
isotropic substances, and later, liquids. The use of this method
is possible because molecules of an isotropic substance, like those
of a liquid, can be considered as minute, individual crystals
which produce interference circles in the beam of a bright-line
X-ray spectrum, although the small number of atoms will cause a reduced sharpness in the circles.

X-ray analyses of such liquids as benzene, hexane, and alcohols revealed an interference pattern of circles, the dimensions of which generally corresponded to theoretical expectations. For benzene in particular these investigations confirm with some accuracy the assumption of chemists, according to whom benzene molecules have six carbon atoms arranged in a circle. Such a circular arrangement is analogous to that of the ice crystal mentioned above. An investigation of water by the same method resulted in a similar sharp interference pattern of circles, demonstrating that some water molecules are oriented in the same way as those in crystals.

Later, similar experiments with liquids were conducted by Kees, who proceeded on the assumption that all molecules are spaced at regular intervals. He showed that by proceeding in this manner the results obtained are in good agreement with theoretical expectations. His calculations were based on density and molecular weight of the liquid according to the following formula:

\[ a_{\text{calc.}} = 1.33 \sqrt{\frac{3M}{d}} \]  

(equ. 6)

The results thus obtained for liquids and liquified gases are given in Table 3. In the second column are listed values for

<table>
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<th>Table 3. Key:</th>
<th>1-Liquid</th>
<th>2-Oxygen</th>
<th>3-Argon</th>
<th>4-Benzene</th>
<th>5-Water</th>
<th>6-Ethyl alcohol</th>
<th>7-Ethyl ether</th>
<th>8-Fumaric acid</th>
<th>9-observed</th>
<th>10-calculated</th>
<th>11-AU</th>
</tr>
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</table>

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the angle \( \psi \) at which the interference-pattern circles were observed. The third and fourth columns give the distances between molecules as determined from the circle dimensions and formula respectively.

It is noteworthy that, in the case of water, a second interference-pattern circle was observed at an angle \( \psi \) of 46°. This same angle was definitely observed, although more faintly, for both liquid oxygen and argon. This demonstrated the presence in liquids of double molecules, separated by the closer spacing of \( a=2.4 \) AU. Such a polymerization demonstrated the polar characteristic of water.

Similar, more recent experimental investigations on the structure of liquids by Debye, Pernik, and Prins were thus provided with a firmer theoretical foundation. The results of these investigations were published almost simultaneously in 1927 in German physics journals.

There is no doubt that the atoms not only of crystals but also of liquids and gaseous substances occupy definite locations in the molecules. Some variations in spacing due to thermal oscillation of the atoms must be allowed. These variations are not large, however, since they probably do not exceed second-order values. There is no basis for assuming that atoms in liquid molecules appear other than in crystalline form - the same form which constituted the structure of molecules. It is expected that an X-ray analysis of gases and vapors, scheduled for the near future, will yield especially reliable results.
According to Bregz, water in the solid state consists of coupled, six-sided rings, each having six oxygen atoms and 12 hydrogen atoms. Armstrong, on the other hand, proposes that liquid water consists of associated molecules, also in the form of six-sided rings similar to those of benzene. Bridgman concludes from his experiments at high pressures that the crystalline orientation of some atoms of water is evident, inasmuch as sharp interference-pattern circles were observed during X-ray analysis.

With this state of affairs, the idea suggests itself that Bregz's firmly-established six-sided links in the lattice of ice, which consist of six oxygen and 12 hydrogen atoms and occupy a large volume, and, therefore, form a less dense structure, are simply those same complex, ice-forming molecules in water described by Tammann, Duclaux, and Armstrong. There is no direct evidence as to these two complexes being identical, but there is much data indicating the great probability of such an assumption. Future investigations will undoubtedly resolve this question, important for understanding the structure and properties of water.
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Table 1

Figure 1

Figure 2

Figure 3

Figure 4
**Table 3**

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$\theta = 3$