

$$\frac{\text{Reading of sample} - \text{reading of blank}}{\text{Reading of standard} - \text{reading of blank}} \times 0.5 \times 10^{-6}$$

Normally the blank with monosaccharide will give nearly the same value as a water blank, and an extraordinarily high monosaccharide blank may be due to a too old hypobromite reagent.

In Table 1 are shown some examples of determinations. The amount of monosaccharide corresponds to different degrees of hydrolysis of the disaccharide. The blank corresponds to 100 % of hydrolysis. It is also possible to have greater amounts of monosaccharide (2–3 times the amounts used in the examples). In determining, e.g., lactose the blank should contain half glucose and half galactose.

Ketohexoses will not be fully oxidized under the conditions given; e.g. fructose will need an oxidation time of at least 1 hour to give a negative anthrone reaction. This difference in oxidation rate has been used for the determination of fructose in the presence of glucose by Slein and Schneel<sup>4</sup>.

Small amounts of protein and acetate and citrate buffers are without influence on the determination; if necessary the pH must be adjusted to about 10 before the addition of hypobromite.

If a faster method is wanted, one of the less accurate "heat of mixing" anthrone methods may be used<sup>5</sup>.

*Acknowledgements.* We are gratefully indebted to Professor Holger Jørgensen for his interest of this work.

1. Dahlqvist, A. *Hog Intestinal  $\alpha$ -Glucosidases* (Diss.), University of Lund 1960, p. 16.
2. Tauber, H. and Kleiner, I. S. *J. Biol. Chem.* **99** (1932) 249.
3. McCready, R. M., Guggolz, J., Silveira, V. and Owens, H. S. *Anal. Chem.* **22** (1950) 1156.
4. Slein, M. W. and Schnell, G. W. *Proc. Soc. Exptl. Biol. Med.* **82** (1953) 734.
5. Snell, F. D. and Snell, C. T. *Colorimetric Methods of Analysis*, 3rd Ed., D. van Nostrand Company, New York 1953, Vol. III, p. 199.

Received April 14, 1961.

## Preliminary Results of an Electron Diffraction Reinvestigation of Cyclobutane and Cyclopentane

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The molecular structure of cyclobutane and cyclopentane have been reinvestigated by the electron diffraction sector method. However, further refinements and a more extensive analysis of the diffraction data have to be performed before the exact equilibrium conformation of the ring-formed carbon skeletons can be determined. Only some preliminary results therefore will be presented in this note.

As members of a group of molecules characterized by a strained conformation of the carbon skeleton, cyclobutane and cyclopentane have been the object of a series of experimental and theoretical studies<sup>1-16</sup>.

By this investigation we hope to be able to obtain results for the molecular structures which are more extensive and more accurate than those previously published. A comparison of the present results with unpublished structure data obtained by the same diffraction unit in 1956–1957, is also of great interest because it provides an opportunity for estimating the reproducibility of the results obtained by this method.

*Cyclobutane.* By an analysis of the intensity curve and of several radial distribution curves obtained by using different damping functions, the following values for the C–C and C–H bond distances have been obtained:

$$\text{C–C: } 1.548 \pm 0.003 \text{ \AA} \text{ and C–H: } 1.092 \pm 0.010 \text{ \AA}.$$

The values found by the unpublished investigation in 1956–1957 are 1.547, Å and 1.089 Å, respectively.

These results demonstrate that the C–C bond distance in a saturated 4-membered ring is longer than the corresponding distance in the linear paraffins. For ethane the values 1.536 Å<sup>17</sup> and 1.534 Å<sup>18</sup> have been reported. Recent investigations of *n*-butane give the values 1.533 Å<sup>19</sup> and 1.539 Å<sup>20</sup>. Finally by an electron diffraction

investigation of *n*-pentane, *n*-hexane and *n*-heptane Bartell *et al.*<sup>21</sup> have obtained the values 1.531 Å, 1.533 Å and 1.534 Å, respectively.

On the other hand our value for the C—C bond distance is significantly shorter than the corresponding value published by Dunitz and Schomaker<sup>1</sup>.

The carbon ring is definitely found to be puckered. However, the magnitude of the deviation from planarity is not determined with any reasonable accuracy at this stage of the investigation. Furthermore, the question of the existence of an "inversion flopping" of the carbon ring has not yet been settled unambiguously.

The values for the bond distances reported here are not expected to be changed significantly by further refinements.

*Cyclopentane.* A study similar to that performed for *cyclobutane* gave the following values for the C—C and C—H bond distances:

C—C:  $1.539 \pm 0.003$  Å and

C—H:  $1.095 \pm 0.010$  Å.

The corresponding values obtained by the above mentioned unpublished investigation are 1.539 Å and 1.089 Å, respectively.

Within the limits of error the C—C bond length in *cyclopentane* therefore is the same as that in the chain paraffins.

The ring system is found to deviate from planarity. The amplitude of the puckering, however, has not been determined. By further refinements we hope to solve this problem and also to study the claimed presence of a pseudo-rotation in the ring<sup>10,13,15</sup>.

1. Dunitz, J. D. and Schomaker, V. *J. Chem. Phys.* **20** (1952) 1703.
2. Rathjens, G. W., Jr., Freeman, N. K., Gwinn, W. D. and Pitzer, K. S. *J. Am. Chem. Soc.* **75** (1953) 5634.
3. Rathjens, G. W., Jr. and Gwinn, W. D. *J. Am. Chem. Soc.* **75** (1953) 5629.
4. Hassel, O. and Viervoll, H. *Tidsskr. Kjem., Bergvesen Met.* **6** (1946) 31.
5. Hassel, O. and Viervoll, H. *Acta Chem. Scand.* **1** (1947) 149.
6. Pauling, L. and Brockway, L. O. *J. Am. Chem. Soc.* **59** (1937) 1223.
7. Tschamler, H. and Voetter, H. *Monatsh.* **83** (1952) 1228.
8. Aston, J. G., Schumann, S. C., Fink, H. L. and Doty, P. M. *J. Am. Chem. Soc.* **63** (1941) 2029.
9. Aston, J. G., Fink, H. L. and Schumann, S. C. *J. Am. Chem. Soc.* **65** (1943) 341.
10. Pitzer, K. S. and Donath, W. E. *J. Am. Chem. Soc.* **81** (1959) 3213.
11. Miller, F. A. and Inskeep, R. G. *J. Chem. Phys.* **18** (1950) 1519.
12. McCullough, J. P. *J. Chem. Phys.* **29** (1958) 966.
13. McCullough, J. P., Pennington, R. E., Smith, J. C., Hossenloop, I. A. and Waddington, G. *J. Am. Chem. Soc.* **81** (1959) 5880.
14. Curnutte, B. and Shaffer, W. F. *J. Mol. Spectroscopy*, **1** (1957) 239.
15. Kilpatrick, J. E., Pitzer, K. S. and Spitzer, R. *J. Am. Chem. Soc.* **69** (1947) 2483.
16. Coulson, C. A. and Moffitt, W. E. *Phil. Mag.* **40** (1949) 1.
17. Almenningen, A. and Bastiansen, O. *Acta Chem. Scand.* **9** (1955) 815.
18. Allen, H. C., Jr. and Plyler, E. K. *J. Chem. Phys.* **31** (1959) 1062.
19. Bonham, R. A. and Bartell, L. S. *J. Am. Chem. Soc.* **81** (1959) 3491.
20. Kuchitsu, K. *Bull. Chem. Soc. Japan* **32** (1959) 748.
21. Bonham, R. A., Bartell, L. S. and Kohl, D. A. *J. Am. Chem. Soc.* **81** (1959) 4765.

Received April 7, 1961.