CRYSTAL STRUCTURE OF METHYL 4,6-O- (O) -
BENZYLIDENE-2-CHLORO-2-DEOXY- a-D-IDOPYRANOsidE. By
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The structure was determined from Weissenberg
photographs, the crystals belong to space group P21
with two molecules per unit cell, and a=11.83,
b=12.70, c=44.62Å, β=91.8°. Refinement led to an R
value of 0.061 using visually estimated intensities
 correlated by double slit photographs. The
structure was determined from sharpened Patterson
sections and refined by least squares.

While most previous structure determinations of
sugars show the pyranoid ring in the chair
conformation, the idopyranoside described has an
ideal skew conformation, C6, half-way between
B3,2 and B3,4. This conformation allows all the
non-α substituents to lie in equatorial positions,
generally regarded as the most stable. In
addition, the dioxane ring has a chair conformation
with the phenyl group equatorial.

C6 skew intermolecular H-bond from the pyranose
oxygen to the CH on C3 may further stabilize the
C6,2 conformation in the solid.

HIGHLY SUBSTITUTED DISACCHARIDES WITH
UNUSUAL α -LINKAGES. By H.-J. Schmidt, N.
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We have investigated two highly substituted disacchari-
des, both having unusual glycosidic linkages. The first
2,3,4,6-tetra-O-acetyl-α-D-mannopyranosyl-6-O-acetyl-2-
azido-3,4-di-O-benzyl-2-deoxy-α-D-glucopyranoside (I)
is a manno-trehalose derivative having an α,α(1→1)
gly-
cosidic linkage. The second, 3,4-di-O-acetyl-2,4-diazido-
3,4-di-O-benzyl-2,4-dideoxy-α-D-glucopyranosyl)-1,6-an-
hydro-2,4-diazido-2,4-dideoxy-3-D-glucopyranose (II),
is composed of two diazido glucose residues, of which
one is present in the 3→1,6-anhydro form and therefore
is forced to adopt the 1C4-conformation.

Because of this the α(1→3) linkage connects a 4C4
and a 6C4 pyranosyl chair being investigated for the first
time by X-ray methods. The common feature of both
disaccharides is that the linkage is axial with respect
to both pyranosyl rings and that the conformation
around the glycosidic linkages is very similar (see
Fig.1). This conformation is equal to that of all glu-
co-trehalose structures so far investigated.
Disaccharides which are (1→1)-linked contain the cha-
 racteristic C-O-C-O-C-O-C-sequence. Because of the a
configuration of both rings and an arrangement of the
glycosidic oxygen bonds which is near to (+sc,+sc),
the outer bonds of that sequence are shortened to
1.405 Å (mean value) whereas the outer bonds are
1.436 Å (mean). Both of the chairs of I and the 6C4
chair of II adopt nearly the perfect chair confor-
mation. Like most 1,6 anhydro pyranoses investigated
previously the 1C4 anhydro-bridged chair shows a con-
formation half between 1C4, and E4. This conformation
is caused mainly by the anti-reflex-effect of the
anhydro-bridge and by trans-synular N →O interactions.

09.2-11  CRYSTALLIZATION AND STRUCTURE
DETERMINATION OF TETRAHYDROPYRAN

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Engaged in structure investigations of basic
compounds in carbohydrate chemistry we have
obtained a crystallization product of Tetra-
hydropyran (THP) at a temperature of about
-75° Centigrade, which is suited for single
crystal X-ray diffraction measurements.
The melting point of THP is at -49° Centigrade.
Substance is sealed in a thin-walled glass
capillary; temperature is controlled by the
gas stream method with an intermediary heat-
ing, using liquid nitrogen as the gas stream
source; crystallization is achieved with the
aid of an electronically controlled heated
coil positioned around the capillary; and
measurements are so far done only with film
methods.

A crystal of THP has been found to be cubic
with a lattice constant of a = 10.4 Å. The
number of formular units per unit cell is eight.
A possible space group which includes some
pseudo-symmetry, is P43n. Intensity data
collection and structure determination are in
progress. - This work is supported by the
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gemeinschaft).