Table 3. *Probable hydrogen bonds in bis(cyclohexylammonium)* D-*glucose 6-phosphate trihydrate crystals*

X—H···O	Translation *a b c*	*X*···O (Å)	H···O (Å)	*X—H*···O (°)
O(1)—H(O1)···O(81)	0 0 1	2·733 (5)	1·81 (6)	160 (6)
O(2)—H(O2)···O(*W*6)	1 0 1	2·837 (7)	2·04 (6)	139 (5)
O(3)—H(O3)···O(91)	1 0 1	2·611 (5)	1·74 (5)	150 (5)
O(4)—H(O4)···O(*W*1)	1 0 0	2·706 (5)	1·77 (5)	163 (5)
O(11*A*)—H(O*A*)···O(7)	0 0 0	2·715 (7)	1·77 (8)	169 (8)
O(11*B*)—H(O*B*)···O(7)	0 0 0	2·747 (11)	1·93 (17)	140 (15)
O(21)—H(O21)···O(*W*3)	0 0 0	2·764 (5)	1·92 (5)	146 (5)
O(31)—H(O31)···O(81)	$\bar{1}$ 0 0	2·681 (5)	1·83 (5)	146 (5)
O(41)—H(O41)···O(*W*4)	0 0 $\bar{1}$	2·683 (6)	1·75 (6)	165 (6)
O(*W*1)—H(*W*1)···O(7)	0 0 0	2·720 (5)	1·77 (5)	172 (5)
O(*W*1)—H(*W*11)···O(71)	0 0 1	2·650 (5)	1·69 (5)	174 (5)
O(*W*2)—H(*W*2)···O(7)	0 0 0	2·789 (6)	1·90 (5)	152 (5)
O(*W*2)—H(*W*21)···O(4)	$\bar{1}$ 0 0	2·771 (6)	2·01 (6)	135 (5)
O(*W*3)—H(*W*3)···O(81)	$\bar{1}$ 0 0	2·727 (5)	1·79 (5)	163 (5)
O(*W*3)—H(*W*31)···O(9)	$\bar{1}$ 0 0	2·698 (6)	1·81 (5)	153 (5)
O(*W*4)—H(*W*4)···O(*W*5)	0 0 0	2·750 (5)	1·83 (5)	160 (5)
O(*W*4)—H(*W*41)···O(*W*1)	0 0 0	2·798 (6)	1·84 (6)	173 (5)
O(*W*5)—H(*W*5)···O(8)	$\bar{1}$ 0 0	2·696 (6)	1·87 (5)	143 (4)
O(*W*5)—H(*W*51)···O(21)	0 0 0	2·846 (5)	1·98 (5)	150 (5)
O(*W*6)—H(*W*6)···*W*(2)	0 0 0	2·962 (8)	2·62 (5)	101 (4)
O(*W*6)—H(*W*61)···*W*(3)	0 0 0	2·883 (8)	2·68 (6)	92 (4)
N(6)—H(N61)···O(8)	0 0 0	3·301 (7)	2·31 (4)	174 (4)
N(6)—H(N61)···O(9)	0 0 0	3·046 (6)	2·40 (5)	122 (4)
N(6)—H(N62)···O(21)	1 0 0	3·173 (7)	2·44 (5)	130 (4)
N(6)—H(N62)···O(31)	1 0 0	3·379 (7)	2·54 (5)	141 (4)
N(6)—H(N63)···O(51)	0 0 0	3·202 (7)	2·31 (3)	149 (4)
N(6)—H(N63)···O(61)	0 0 0	2·917 (6)	2·16 (4)	131 (4)
N(7)—H(N71)···O(6)	0 0 0	2·944 (6)	2·18 (5)	132 (4)
N(7)—H(N71)···*W*(2)	0 0 0	3·209 (6)	2·60 (5)	119 (4)
N(7)—H(N72)···O(5)	0 0 0	2·956 (6)	2·05 (3)	150 (4)
N(7)—H(N73)···O(3)	$\bar{1}$ 0 0	2·908 (6)	2·00 (5)	149 (4)
N(7)—H(N73)···O(71)	0 0 1	3·154 (5)	2·69 (5)	108 (3)
N(8)—H(N81)···O(91)	0 0 0	2·694 (6)	1·72 (5)	162 (5)
N(8)—H(N82)···O(2)	$\bar{1}$ 0 $\bar{1}$	3·057 (6)	2·57 (5)	110 (4)
N(8)—H(N82)···O(11*A*)	0 0 0	3·175 (8)	2·42 (5)	132 (4)
N(8)—H(N83)···O(9)	0 0 0	2·794 (6)	1·85 (4)	155 (4)
N(9)—H(N91)···O(8)	0 0 0	2·840 (6)	1·85 (5)	173 (4)
N(9)—H(N92)···O(71)	0 0 1	2·745 (6)	1·77 (4)	164 (4)
N(9)—H(N93)···O(41)	1 0 1	3·026 (6)	2·04 (4)	169 (4)

contrast to the Ba salt, there are no intramolecular H bonds between phosphate O atoms and sugar hydroxyl groups. Also interesting is the fact that the O(11*A*)H and O(11*B*)H hydroxyl groups are involved in hydrogen bonds to the same O(7) atom.

Financial support was received from the RP.II.10 program.

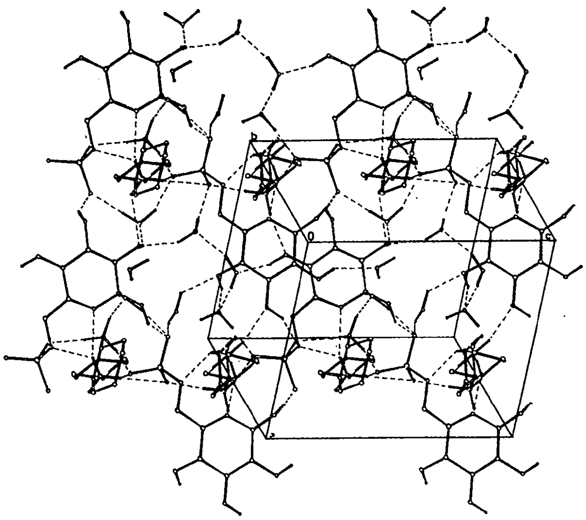

Fig. 2. The packing arrangement. Dashed lines show hydrogen bonds. (C-bonded H atoms are omitted for clarity.)

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Structure of a Novel Hexaaza Macrocyclic Ligand of Schiff-Base Type

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Abstract. 3,7,11,18,22,26-Hexaazapentacyclo[$26.2.2.2^{13,16}.0^{2,7}.0^{17,22}$]tetratriaconta-1(31),11,13(34),14,16(33),26,28(32),29-octaene, $C_{28}H_{38}N_6$, M_r = 458·6, orthorhombic, *Fdd*2, $a = 16{\cdot}328\ (1)$, $b = 56{\cdot}632\ (3)$, $c = 5{\cdot}426\ (1)$ Å, $V = 5017\ (1)$ Å^3, $Z = 8$, $D_x = 1{\cdot}214\ \mathrm{g\ cm^{-3}}$, $\lambda = 1{\cdot}54178$ Å, $\mu(\mathrm{Cu}\ K\alpha) =$

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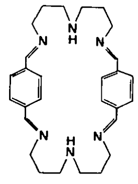
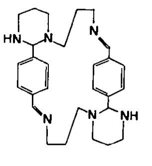
5·385 cm^{-1}, $F(000) = 1984$, $T = 298$ K, final $R = 0·036$ for 1219 reflections. The molecule has a twofold axis coinciding with a crystallographic twofold axis. The molecule exhibits a small molecular cavity open on one side of the macrocyclic ring and closed on the other side.

Introduction. An effort to synthesize the Schiff-base type macrocyclic ligand with four —CH=N— units (I) was made because of the possibility of simultaneous complexation of two transition-metal cations. In the course of preparation of macrocycle (I), formation of the compound with two 1,3-diazacyclohexane rings incorporated into the macrocycle was observed by other spectroscopic methods (Pietraszkiewicz & Gąsiorowski, 1989). The X-ray diffraction analysis of that compound was undertaken in order to establish which of the two possible isomers [(II) or (III)] was obtained.


(I)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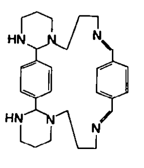
(III)

Experimental. Crystal approximate dimensions 0·18 × 0·28 × 0·28 mm; intensities measured at 298 K on Enraf–Nonius CAD-4 four-circle diffractometer (Cu $K\alpha$ radiation, $\lambda = 1·54178$ Å, graphite monochromator). Lattice parameters determined by least squares from 24 reflections ($16 < \theta < 35°$). Total of 2748 reflections (1317 independent reflections, $R_{\rm int} = 0·017$) up to $\theta = 70°$ measured in the ω–2θ scan mode, index range $h0\rightarrow19$, $k-68\rightarrow68$, $l-6\rightarrow0$. The data were collected in a monoclinic unit cell of C type with $a = 5·431$ (1), $b = 16·328$ (1), $c = 28·473$ (1) Å, $\beta = 95·50°$, and the space group was established to be *Cc*. After structure solution the twofold symmetry of the molecule was detected and the unit cell was transformed to the orthorhombic *Fdd*2 space group; transformation matrix [010, 102, 100] was applied. 1219 reflections considered as observed [$F_o > 2\sigma(F_o)$]. The intensity variation of three standard reflections, measured every hour, was less than 1·5%. No absorption or secondary-extinction correction. Structure solved by *MULTAN*11/82 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1982) and refined with the *SDP* system (B. A. Frenz & Associates Inc., 1985). Weights of each reflection in the refinement (on F) were calculated from $w = 1/\sigma^2(F_o)$, $\sigma(F_o)$ being the e.s.d., based on counting statistics, of the observed structure factor. Scattering factors taken from *International Tables for X-ray Crystallography* (1974). All the H atoms were included in the refinement in calculated positions [d(C—H) = 1·08 Å]. The total number of parameters refined was 173: one scale factor, position parameters and anisotropic thermal parameters for non-H atoms, and isotropic thermal parameters for H atoms. Refinement resulted in final values of $R = 0·036$, $wR = 0·039$ and $S = 2·89$; in the last cycle $(\Delta/\sigma)_{\rm max} = 0·01$. Final max. and min. $\Delta\rho$ were 0·12 and $-0·17$ e Å^{-3}, respectively. All calculations performed on a MicroPDP11/73 computer. The final atomic coordinates are given in Table 1.*

Table 1. *Fractional atomic coordinates* ($\times 10^4$) *and equivalent isotropic thermal parameters* (Å^2) *for non-*H *atoms, with e.s.d.'s in parentheses*

$$B_{\rm eq} = \tfrac{4}{3}[a^2B(1,1) + b^2B(2,2) + c^2B(3,3) + ab(\cos\gamma)B(1,2) + ac(\cos\beta)B(1,3) + bc(\cos\alpha)B(2,3)].$$

	x	y	z	$B_{\rm eq}$
C(1)	5996 (1)	318·0 (3)	1639	3·35 (4)
N(2)	6123 (1)	479·6 (3)	101 (5)	3·69 (4)
C(3)	5838 (1)	717·9 (3)	740 (6)	3·58 (5)
C(4)	5403 (1)	829·1 (3)	−1447 (5)	3·43 (4)
C(5)	4538 (1)	734·1 (3)	−1865 (5)	3·17 (4)
N(6)	3968 (1)	811·1 (2)	84 (4)	2·99 (3)
C(7)	3737 (1)	1060·3 (3)	−341 (5)	3·78 (5)
C(8)	3172 (1)	1149·3 (3)	1642 (6)	4·10 (5)
C(9)	2426 (1)	990·2 (3)	1831 (7)	4·58 (6)
N(10)	2714 (1)	748·1 (3)	2205 (5)	3·92 (4)
C(11)	3211 (1)	667·4 (3)	136 (5)	3·12 (4)
C(12)	3403 (1)	407·7 (3)	530 (5)	2·89 (4)
C(13)	3810 (1)	335·6 (3)	2638 (5)	3·50 (4)
C(14)	3986 (1)	97·7 (3)	2981 (5)	3·41 (4)
C(15)	3762 (1)	−69·6 (3)	1231 (5)	3·14 (4)
C(16)	3335 (1)	2·2 (3)	−847 (5)	3·34 (4)
C(17)	3154 (1)	240·4 (3)	−1195 (5)	3·18 (4)

Discussion. The isomer found in the course of the crystal structure determination was (II). The bond lengths, bond angles and selected torsion angles are given in Table 2. The molecule has a twofold axis coinciding with the crystallographic twofold axis. The diazacyclohexane six-membered ring has a chair conformation with the average atom deviation from the r.m.s. plane equal to 0·246 (3) Å [range from −0·214 (3) Å for C(8) to +0·274 (3) Å for C(11)]. The six atoms of the aromatic ring are planar within 0·014 (2) Å [average deviation from planarity 0·009 (2) Å]. The conformation of the 22-membered central macrocyclic ring is described by torsion angles (Table 2) and is illustrated in Fig. 1. The two aromatic rings and the two diazacyclohexane rings are inclined to the least-squares plane of the central macrocyclic ring by the angles 61·5 (1) and 29·8 (1)°,

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* Lists of structure factors, anisotropic thermal parameters of non-H atoms, positional and isotropic thermal parameters of H atoms, and shortest non-bonded contacts have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52177 (17 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 2. *Bond distances* (Å), *bond angles* (°) *and selected torsion angles* (°) *for non*-H *atoms, with e.s.d.'s. in parentheses*

C(1)—N(2)	1·256 (3)	N(10)—C(11)	1·458 (3)
N(2)—C(3)	1·469 (2)	C(11)—C(12)	1·519 (2)
C(3)—C(4)	1·519 (4)	C(12)—C(13)	1·384 (4)
C(4)—C(5)	1·529 (3)	C(12)—C(17)	1·392 (3)
C(5)—N(6)	1·474 (4)	C(13)—C(14)	1·390 (3)
N(6)—C(7)	1·480 (2)	C(14)—C(15)	1·390 (3)
N(6)—C(11)	1·480 (2)	C(15)—C(16)	1·387 (4)
C(7)—C(8)	1·505 (4)	C(15)—C(1^{i})	1·478 (3)
C(8)—C(9)	1·519 (4)	C(16)—C(17)	1·395 (3)
C(9)—N(10)	1·464 (3)		

C(1)—N(2)—C(3)	117·4 (2)	N(10)—C(11)—C(12)	108·1 (2)
N(2)—C(3)—C(4)	110·1 (2)	C(11)—C(12)—C(13)	120·0 (2)
C(3)—C(4)—C(5)	113·7 (2)	C(11)—C(12)—C(17)	120·3 (3)
C(4)—C(5)—N(6)	111·9 (2)	C(13)—C(12)—C(17)	119·7 (2)
C(5)—N(6)—C(7)	109·3 (2)	C(12)—C(13)—C(14)	119·7 (2)
C(5)—N(6)—C(11)	112·2 (2)	C(13)—C(14)—C(15)	121·0 (2)
C(7)—N(6)—C(11)	108·3 (1)	C(14)—C(15)—C(16)	119·2 (2)
N(6)—C(7)—C(8)	111·4 (3)	C(14)—C(15)—C(1^{i})	118·4 (2)
C(7)—C(8)—C(9)	110·0 (3)	C(16)—C(15)—C(1^{i})	122·4 (2)
C(8)—C(9)—N(10)	107·9 (2)	C(15)—C(16)—C(17)	120·0 (2)
C(9)—N(10)—C(11)	111·5 (3)	C(12)—C(17)—C(16)	120·4 (2)
N(6)—C(11)—N(10)	107·9 (2)	C(15)—C(1^{i})—N(2^{i})	123·4 (2)
N(6)—C(11)—C(12)	111·3 (1)		

C(1)—N(2)—C(3)—C(4)	−134·6 (2)	C(11)—C(12)—C(17)—C(16)	179·5 (2)
N(2)—C(3)—C(4)—C(5)	76·3 (2)	C(12)—C(13)—C(14)—C(15)	0·1 (3)
C(3)—C(4)—C(5)—N(6)	69·8 (2)	C(13)—C(14)—C(15)—C(1^{i})	176·9 (2)
C(4)—C(5)—N(6)—C(11)	−163·0 (2)	C(1^{i})—C(15)—C(16)—C(17)	−177·1 (2)
C(5)—N(6)—C(11)—C(12)	58·4 (3)	C(14)—C(15)—C(1^{i})—N(2^{i})	−175·4 (1)
N(6)—C(11)—C(12)—C(13)	59·9 (3)	C(16)—C(15)—C(1^{i})—N(2^{i})	3·4 (2)
N(6)—C(11)—C(12)—C(17)	−121·6 (2)	C(15)—C(16)—C(17)—C(12)	0·3 (3)
C(11)—C(12)—C(13)—C(14)	−179·7 (2)	C(15)—C(1^{i})—N(2^{i})—C(3^{i})	179·1 (1)

Symmetry code: (i) $1-x,\ -y,\ z$.

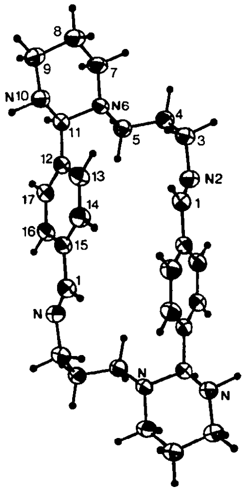

Fig. 1. *ORTEP*II (Johnson, 1976) view of the molecule with the atom numbering. The thermal ellipsoids are given at the 50% probability level.

respectively. The dihedral angle between the two symmetry related aromatic rings is 122·9° and that between the two diazacyclohexane rings is 59·5 (1)°. The r.m.s. planes of the aromatic ring and the diazacyclohexane ring joined by the C(11)—C(12) bond are perpendicular to each other [90·3 (1)°]. The molecule is elongated in the C(5)···C(5^{i}) [symmetry code (i) = $1-x,\ -y,\ z$] direction [d = 8·451 (2) Å]; the shortest intramolecular contact across the macrocyclic ring occurs between the atoms C(14) and C(14^{i}) and is equal to 3·491 Å [H(14)···H(14^{i}) distance is 2·32 Å]. Consequently, the molecule exhibits a small internal cavity of approximate dimensions 4 Å length, 2 Å width and 1 Å depth, which is open on the C(16)—C(17)—C(16^{i})—C(17^{i}) side and closed, by the H(14)···H(14^{i}) short intramolecular contact, on the other side. The cavity dimensions were estimated by tracing spheres at van der Waals radii about the atoms in the molecule and measuring the 'empty' space (Fig. 2*a* and *b*). The cavity is 'filled' by the H(14) and H(14^{i}) atoms which belong to the neighbouring molecule translated by the vector [001] (Fig. 2*c*); the penetration of the internal cavity is less than 1 Å in depth. Because the dimensions of the internal cavity are so small it is unlikely that the investigated macrocyclic molecule is able to form inclusion-type complexes.

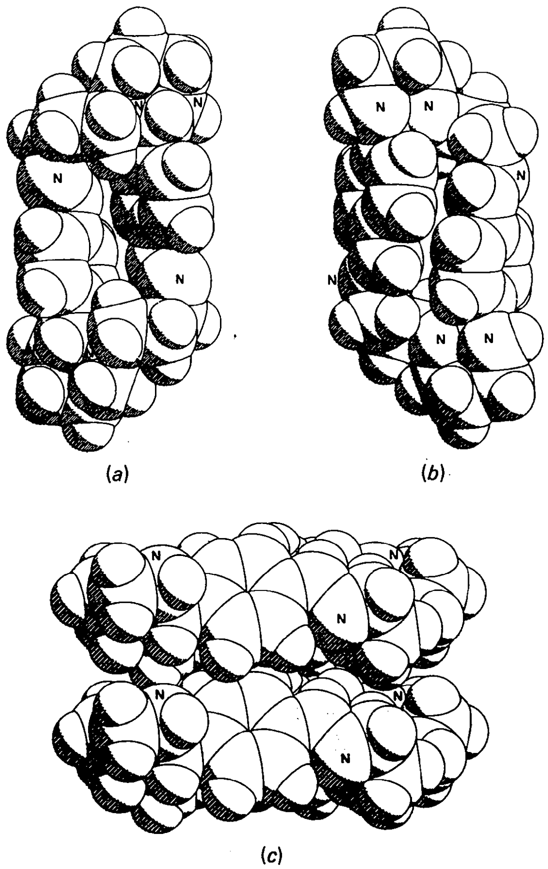

Fig. 2. Space-filling representation of the molecule: (*a*) view of the cavity, (*b*) bottom of the cavity showing the H(14)···H(14^{i}) intramolecular interaction, and (*c*) penetration of the internal cavity by the neighbouring molecule translated by the vector [001].

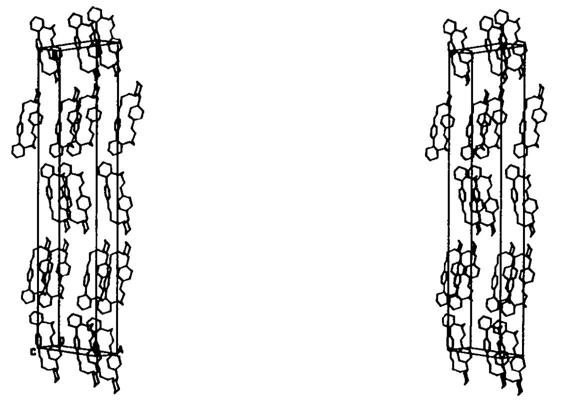

Fig. 3. Packing diagram; H atoms are omitted for clarity.

Fig. 3 shows how the molecules pack in the crystal. The shortest interatomic distances to adjacent different molecules are: no C···C or C···N below 3·5 Å; N···N 3·396 (3) Å, between N(2) and N(10) at $\frac{1}{2}+x, y, -\frac{1}{2}+z$; N···H 2·44 Å, between N(2) and H(10) at $\frac{1}{2}+x, y, -\frac{1}{2}+z$; C···H 2·86 Å, between C(5) and H(13) at $x, y-1+z$; and H···H 2·29 Å, between H(3*a*) and H(9*b*) at $\frac{1}{2}+x, y, -\frac{1}{2}+z$.

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2-(*p*-Methoxybenzoylmethylene)-3-phenyl-1,3-thiazolidine-4,5-dione, a Compound with a C—S···O Interaction

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Abstract. $C_{18}H_{13}NO_4S$, $M_r = 339{\cdot}4$, monoclinic, $P2_1/n$, $a = 8{\cdot}359$ (1), $b = 9{\cdot}961$ (2), $c = 19{\cdot}048$ (3) Å, $\beta = 93{\cdot}749$ (12)°, $V = 1582{\cdot}83$ Å^3, $Z = 4$, $D_m = 1{\cdot}43$, $D_x = 1{\cdot}42$ Mg m^{-3}, λ(Cu $K\alpha$) = 1·54178 Å, $\mu = 1{\cdot}897$ mm^{-1}, $F(000) = 704$, $T = 293$ K, $R = 0{\cdot}0508$ for 2390 unique observed reflections. The molecule has a short intramolecular S···O contact of 2·697 Å and a C—S···O angle of 164·8°.

Introduction. This study is supplementary to an earlier one (Ciechanowicz-Rutkowska, Grochowski & Stec, 1989) and concerns the structure of the Knoevenagel reaction substrate, crystallized successfully in a form of a *p*-methoxybenzoyl derivative, while the earlier study concerned the structure of the reaction product. This work was undertaken in order to assess the role of the benzoyl substituent in the course of the reaction with malonodinitrile which takes place for compound *A* but not for compound *B*, as shown schematically in Fig. 1.

Experimental. The title compound was obtained by Dr J. Eilmes (Żankowska-Jasinska & Eilmes, 1973) and crystallized from chlorobenzene at room tem-

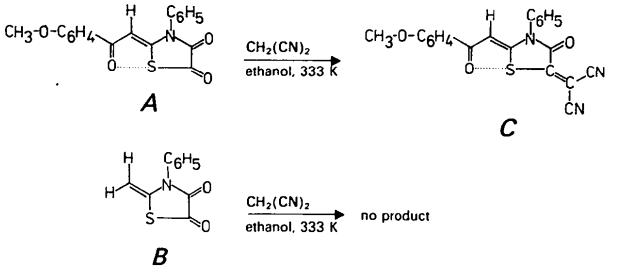

Fig. 1. The Knoevenagel reaction.

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