Preparation of Bicyclo[3.2.0]heptane-2-*endo*,7-*endo*-diols: 1,3-Diols with a Chiral Rigid Backbone.

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SUPPORTING INFORMATION

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General: ^1H NMR spectra were recorded in CDCl_3 at 300.00 MHz at 20 °C with either tetramethylsilane (δ 0.00) or chloroform (δ 7.26) as the internal standard. ^{13}C NMR spectra were recorded in CDCl_3 at 75.46 MHz at 20 °C with chloroform (δ 77.0) as the internal standard. Signal multiplicities were established by DEPT experiments. Melting points were determined through a *Büchi* instrument and are uncorrected. Specific optical rotations were determined at the D line through a *Perkin Elmer 341* polarimeter. Flash chromatographic separations were performed with a Gilson MPLC system over Merck Silica gel 60 (230-400 mesh), tlc analyses were performed over Merck precoated tlc plates (Silica gel 60 GF₂₅₄ 0.25 mm). Tetrahydrofuran was obtained anhydrous over sodium and benzophenone. Unless otherwise stated, other solvents and reagents were used as commercially available.

Carbonyl acid tert-butyl ester 2,2-dimethyl-bicyclo[3.2.0]hept-3-en-6-yl ester (6a). The reaction was completed after 2.5h. An analytical sample was obtained after flash chromatography (PE:Et₂O 40:1, R_f 0.25, Ce^{iv}) as a colourless oil. GC (T_i) R_{t(min)}: (50°C) 10.63. ¹H NMR δ (ppm): 5.54 (d, 1H, J = 5.77 Hz), 5.44 (dd, 1H, J₁ = 5.49 Hz, J₂ = 12.2 Hz), 4.78-4.70 (m, 1H), 3.65 (m, 1H), 2.28-2.14 (m, 1H), 2.05-1.95 (m, 2H), 1.40 (s, 9H), 0.98 (s, 3H), 0.88 (s, 3H). ¹³C NMR δ (ppm): 152.8, 144.3, 125.7, 81.5, 68.9, 52.3, 47.7, 40.1, 29.8, 29.3, 27.7, 20.4. I.R. (neat) ν (cm⁻¹): 2953, 1737, 1369, 1278, 1168, 1108. m/z: 138 (<1%), 120 (<1%), 105 (20%), 94 (48%), 79 (100%), 57 (78%), 44 (49%). Anal. calcd for C₁₄H₂₂O₃ C, 70.56; H, 9.30. Found: C, 70.48; H, 9.33.

Carbonyl acid tert-butyl ester 4-methyl-bicyclo[3.2.0]hept-3-en-6-yl ester (6b). The reaction was completed after 2.25 h. An analytical sample was obtained after flash chromatography (Pentane +1%Et₂O, R_f 0.24, Ce^{iv}, yield 74%) as a white solid m.p. 35-37°C. GC (T_i) R_{t(min)}: (50°C) 10.43. ¹H NMR δ (ppm): 5.72 (bs, 1H), 5.30 (q, 1H, J = 7.8 Hz), 3.75 (bs, 1H), 2.88-2.62 (m, 3H), 2.35-2.32 (m, 1H), 2.14 (dt, 1H, J = 11.5, 8.1 Hz), 2.02 (s, 3H), 1.73 (s, 6H). ¹³C NMR δ (ppm): 152.9, 138.7, 127.4, 81.6, 71.9, 55.3, 40.1, 34.7, 29.5, 27.8, 16.4. I.R. (KBr) ν (cm⁻¹): 2920, 1739, 1367, 1280, 1164, 1107, 864. m/z: 41 (78%), 57 (70%), 80 (100%), 91 (45%), 122. Anal. calcd for C₁₃H₂₀O₃: C, 69.61; H, 8.99. Found: C, 69.40; H, 9.09.

Carbonyl acid tert-butyl ester 1,4-dimethyl-bicyclo[3.2.0]hept-3-en-6-yl ester (6c). The reaction was completed after 1 h. An analytical sample was obtained after flash chromatography (Pentane +2%Et₂O, R_f 0.28, Ce^{iv}, yield 60%) as a colourless oil. GC (T_i) R_{t(min)}: (50°C) 10.39. ¹H NMR δ (ppm): 5.38 (t, 1H), 5.06 (dd, 1H, J = 15.7, 8.2 Hz), 3.02 (m, 1H), 2.21-2.11 (m, 3H), 2.00 (dd, 1H, J = 11.8, 8.2 Hz), (m, 3H), 1.40 (s, 9H), 1.10 (s, 3H). ¹³C NMR δ (ppm): 153.1, 139.2, 127.5, 81.8, 71.8, 59.2, 48.1, 40.2, 38.2, 27.9, 24.6, 16.7. IR (neat) ν (cm⁻¹): 2980, 1740,

1368, 1281, 1156, 1100, 867. m/z: 238 (<1%), 121 (13%), 94 (72%), 57 (100%). Anal. calcd for C₁₄H₂₂O₃: C, 70.56; H, 9.30. Found: C, 70.41; H, 9.27.

Carbonic acid bicyclo[3.2.0]hept-3-en-6-yl ester tert-butyl ester (6d). The reaction was completed after 1h and 10'. An analytical sample was obtained after flash chromatography (PE:Et₂O 35:1, R_f 0.21, Ce^{iv}, yield 76%) as a colourless oil. GC (T_i) R_{t(min)}: (50°C) 9.82. ¹H NMR δ (ppm): 5.85 (m, 1H), 5.62 (m, 1H), 4.90 (q, 1H, J = 7.6 Hz), 3.65 (m, 1H), 2.58-2.35 (m, 3H), 2.14 (ddd with further fine couplings, 1H, J = 16.5, 5.5, 2.5 Hz), 1.76 (dt with further fine couplings, 1H, J = 11.5, 7.7 Hz), 1.42 (s, 9H). ¹³C NMR δ (ppm): 152.9, 133.7, 128.7, 81.8, 72.1, 53.1, 40.7, 34.8, 28.7, 27.9. IR (neat) ν (cm⁻¹): 2981, 1735, 1280, 1256, 1164, 1108. m/z: 195 (<1%), 110 (11%), 93 (60%), 66 (57%), 57 (100%), 41 (100%). Anal. calcd. for C₁₂H₁₈O₃: C, 68.54; H, 8.63. Found: C, 68.44; H, 8.63.

Carbonic acid tert-butyl ester 1-methyl-bicyclo[3.2.0]hept-3-en-6-yl ester (6e). The reaction was completed after 1h and 45'. The product was obtained as a yellow oil and was not purified further. (Pentane +1%Et₂O, R_f 0.21, Ce^{iv}). GC (T_i) R_{t(min)}: (50°C) 9.86. ¹H NMR δ (ppm): 5.79 (m, 1H), 5.58 (m, 1H), 4.97 (q, 1H, J = 7.7 Hz), 3.10 (m, 1H), 2.27-2.13 (m, 3H), 1.95 (m, 1H), 1.20 (s, 9H), 1.10 (s, 3H). ¹³C NMR δ (ppm): 153.0, 133.7, 129.2, 81.7, 71.7, 56.9, 48.3, 40.3, 37.5, 27.8, 24.3. IR (neat) ν (cm⁻¹): 2980, 1736, 1368, 1278, 1165, 1153, 1109, 864. m/z: 223 (<1%), 209 (<1%), 123 (3%), 91 (12%), 80 (48%), 57 (100%), 41 (35%). Anal. calcd for C₁₃H₂₀O₃: C, 69.61; H, 8.99. Found: C, 69.70; H, 9.02.

Carbonic acid 2-benzyloxymethyl-bicyclo[3.2.0]hept-3-en-6-yl ester tert-butyl ester (6f). The reaction was completed after 2h. An analytical sample of the 2-*exo*:2-*endo* mixture (3:1) was obtained after flash chromatography (Pentane +1%Et₂O, R_f 0.17, UV and Ce^{iv}, yield 70%) as a colourless oil. ¹H NMR (major isomer) δ (ppm): 7.40-7.20 (m, 5H), 5.87 (m, 1H), 5.78 (m, 1H),

4.96 (q, 1H, J = 7.8 Hz), 4.48 (s, 2H), 3.70 (m, 1H), 3.31 (dd, 1H, J = 9.1, 5.8 Hz), 3.13 (dd, 1H, J = 9.1, 8.0 Hz), 2.78 (m, 1H), 2.71-2.52 (m, 1H), 2.37 (dd, 1H, J = 13.7, 7.1 Hz), 1.85 (dt with further fine couplings, 1H, J = 12.3, 7.5 Hz), 1.44 (s, 9H). ^1H NMR (minor isomer) δ (ppm): 7.40-7.20 (m, 5H), 5.73 (m, 2H), 4.87 (q, 1H, J = 7.7 Hz), 4.52 (d, 1H, J = 12.1 Hz), 4.46 (d, 1H, J = 12.1 Hz), 3.70 (m, 1H), 3.57 (dd, 1H, J = 9.1, 6.9 Hz), 3.47 (t, 1H, J = 8.9 Hz), 3.22 (m, 1H), 2.71-2.52 (m, 1H), 2.32-2.19 (m, 1H), 2.15-2.03 (m, 1H). ^{13}C NMR (major isomer) δ (ppm): 152.8, 138.5, 134.7, 130.7, 128.4, 127.6, 81.8, 73.1, 73.1, 71.7, 54.3, 52.2, 33.9, 32.0, 27.8. ^{13}C NMR (minor isomer) δ (ppm): 152.8, 138.4, 134.4, 129.8, 128.4, 127.7, 81.8, 73.2, 70.0, 68.9, 52.9, 49.3, 31.3, 28.2, 27.8. IR (neat) ν (cm^{-1}): 2979, 1735, 1367, 1278, 1164, 1109, 738, 698. m/z: 329 (<1%), 186 (5%), 121 (12%), 91 (86%), 57 (100%). Anal. calcd for $\text{C}_{20}\text{H}_{26}\text{O}_4$: C, 72.70; H, 7.93; O, 19.37. Found: C, 72.57; H 7.94.

3-Iodo-2,2-dimethyl-hexahydro-4,6-dioxo-cyclobuta[cd]inden-5-one (7a). The reaction of iodocarbonation was completed after 30 min. A pale yellow solid was obtained (90% yield). GC (T_i) $R_{t(\text{min})}$: (50°C) 15.664. After treatment with Et_2O a white solid was obtained (62% yield) (turns brown at 96-98°C melts at 131°C). Et_2O , R_f 0.5, UV and Ce^{iv} . ^1H NMR δ (ppm): 4.98 (t, with further fine couplings 1H, J = 9.46 Hz), 4.92 (td with further fine couplings, 1H, J = 7.7, 5.0 Hz), 4.31 (d, 1H, J = 9.6 Hz), 3.12 (m, 1H), 2.63 (dtd, 1H, J = 14.6, 8.9, 2.5 Hz), 2.45 (m, 1H), 1.90 (dt, 1H, J = 14.6, 5.4 Hz), 0.95 (s, 3H), 0.90 (s, 3H). ^{13}C NMR δ (ppm): 146.8, 87.6, 69.3, 44.9, 39.7, 39.6, 34.2, 30.7, 27.5, 20.3. IR (KBr) ν (cm^{-1}): 2963, 1750, 1464, 1386, 1255, 1172, 1089, 814, 773. m/z: 308 (2%), 137 (15%), 127 (10%), 91 (65%), 79 (40%), 69 (100%). Anal calcd for $\text{C}_{10}\text{H}_{13}\text{IO}_3$: C, 38.98; H, 4.25. Found: C, 39.12; H 4.27.

3-Iodo-4,4-dimethyl-bicyclo[3.2.0]heptane-2,7-diol (8a). The reaction was complete after 48 h. After flash chromatography (PE:Et₂O 1:1; R_f 0.19, UV and Ce^{iv}) the product was obtained as a white wax (60% yield). ¹H NMR (CD₃OD) δ (ppm): 4.80-4.65 (m, 3H), 2.95 (q with further fine couplings, 1H, J₁ = 7.6 Hz), 2.60 (dtd, 1H, J₁ = 13.2, 8.8, 2.7 Hz), 2.22 (m, 1H), 1.82 (m, 1H), 0.95 (s, 3H), 0.90 (s, 3H). ¹³C NMR (CD₃OD) δ (ppm): 85.0, 66.4, 51, 43.8, 43.2, 39.2, 34.3, 28.5, 21.8. IR (KBr) ν (cm⁻¹): 3275, 2949-2892, 2361, 1436, 1364, 1237, 1089, 1072. m/z: 283 (<1%), 264 249 (1%), 235 (<1%), 221 (28%), 137 (65%), 119 (25%), 109 (72%), 95 (86%), 55 (58%), 43 (93%), 39 (100%). Anal. calcd for C₉H₁₅IO₂: C, 38.32; H, 5.36. Found C, 38.26; H 5.35.

3-Iodo-2-methyl-bicyclo[3.2.0]heptane-2,7-diol (8b). The reaction of iodocarbonation was completed after 1 h and the crude product obtained was not further purified. The acid hydrolysis was completed after 30 h and led quantitatively to an orange solid. An analytical sample was obtained by flash chromatography (DCM:Et₂O 5:1; R_f 0.43, UV and Ce^{iv}, yield 66%) as a yellow solid (m.p. 84-86 °C). ¹H NMR δ (ppm): 4.85 (dd, 1H, J = 11.8, 7.0 Hz), 4.56 (bs shifting, 2H), 4.51 (m, 1H), 2.64 (t with further fine couplings, 1H, J = 7.6 Hz), 2.57 (dtd, 1H, J = 13.5, 8.8, 1.9 Hz), 2.31 (m, 1H), 2.19-2.00 (m, 2H), 1.64 (dtd, 1H, J = 13.5, 5.5, 1.1 Hz), 1.20 (s, 3H). ¹³C NMR δ (ppm): 83.0, 65.9, 45.1, 43.6, 38.9, 36.6, 30.6, 29.1. IR (KBr) ν (cm⁻¹): 3278, 3207, 2976, 1444, 1264, 1203, 1158, 1108, 1007, 934, 628. m/z: 268 (<1%), 127 (100%), 123, 80 (14%), 63 (10%), 41 (16%). Anal. calcd for C₈H₁₃IO₂: C, 35.84; H, 4.89. Found: C, 35.81; H, 4.89.

3-Iodo-2,5-dimethyl-bicyclo[3.2.0]heptane-2,7-diol (8c). The reaction was completed after 24 h. A waxy solid was obtained (93% yield) that was purified by flash chromatography (DCM:Et₂O 20:1; R_f 0.20, UV and Ce^{iv}, yield 67%) as a white solid (turns brown at 88 °C, melts

at 96 °C). ^1H NMR δ (ppm): 4.90 (dd, 1H, $J = 12.9, 6.6$ Hz), 4.58 (ddd, 1H, $J = 14.0, 8.3, 5.8$ Hz), 4.05 (bs, 2H), 2.28 (d with further fine couplings, 1H, $J = 8.3$ Hz), 2.21 (ddd, 1H, $J = 13.2, 9.1, 1.7$ Hz), 2.12 (dd, 1H, $J = 12.9, 6.6$ Hz), 1.89 (dd, 1H, $J = 13.2$ Hz), 1.84 (ddd, 1H, $J = 13.5, 5.8, 1.1$ Hz), 1.22 (s, 3H), 1.08 (s, 3H). ^{13}C NMR δ (ppm): 82.6, 63.6, 50.6, 50.4, 42.7, 39.3, 38.4, 29.2, 26.7. IR (KBr) ν (cm^{-1}): 3256, 2935, 1451, 1203, 1109, 649. m/z : 221 (19%), 137 (36%), 111 (38%), 93 (43%), 87 (79%), 43 (100%). Anal. calcd for $\text{C}_9\text{H}_{15}\text{IO}_2$: C, 38.32; H, 5.36. Found: C, 38.21; H 5.32.

3-Iodobicyclo[3.2.0]heptane-2,7-diol (8d). ^1H NMR δ (ppm): 4.63-4.46 (m, 2H), 4.35-4.28 (t, 1H, $J = 8.7$ Hz), 4.15 (bs, 2H), 2.82 (q, 1H, $J = 7.8$ Hz), 2.57 (dtd, 1H, $J = 13.5, 8.8, 2.2$ Hz), 2.30 (m, 1H), 2.13 (m, 2H), 1.62 (dtd, 1H, $J = 13.2, 6.0, 1.1$ Hz). ^{13}C NMR δ (ppm): 85.5, 65.9, 43.2, 42.3, 36.7, 31.1, 30.7. IR (KBr) ν (cm^{-1}): 3342, 2971, 1443, 1261, 1222, 1173, 1101, 1068, 767, 735. m/z : 254 (8%), 193 (79%), 109 (100%), 81 (89%), 73 (79%), 66 (60%), 55 (60%), 41 (54%). Anal. calcd for $\text{C}_7\text{H}_{11}\text{IO}_2$: C, 33.09; H, 4.36. Found: C, 32.99; H, 4.36.

3-Iodo-5-methyl-bicyclo[3.2.0]heptane-2,7-diol (8e). TLC (DCM: Et_2O 20:1), R_f 0.2 (UV visible). Mp: 84-86 °C (DCM:pentane = 1:1). ^1H NMR δ (ppm): 4.63-4.50 (m, 2H), 4.33 (t, 1H, $J = 9.1$ Hz), 4.20 (bs, 2H), 2.38 (td, 1H, $J = 8.1, 1.4$ Hz), 2.25-2.10 (ddd superimposed with a dd, 2H, $J = 11.5, 9.1, 2.5$ Hz, $J = 12.9, 6.3$ Hz), 1.92-1.74 (dd superimposed with a dd, 2H, $J = 11.8$ Hz and $J = 13.5, 6.0$ Hz), 1.15 (s, 3H). ^{13}C NMR δ (ppm): 85.3, 64.1, 50.3, 47.4, 42.7, 38.7, 31.4, 26.2. IR (film), ν_{max} (cm^{-1}): 3263, 2950, 1278, 1164, 1102, 1058. m/z : 268 (<1%), 207 (72%), 141 (9%), 123 (33%), 105 (29%), 97 (85%), 93 (87%), 80 (65%), 57 (53%), 41 (100%). Anal calcd for $\text{C}_8\text{H}_{13}\text{IO}_2$: C, 35.84; H, 4.89. Found: C, 35.98; H, 4.93.

4-Benzylloxymethyl-3-iodo-bicyclo[3.2.0]heptane-2,7-diol (8f). TLC (DCM: Et_2O 10:1), R_f 0.3 (UV visible, Ce (IV)). *Major isomer.* ^1H NMR δ (ppm): 7.40-7.20 (m, 5H); 4.82-4.70 (m,

1H); 4.70-4.65 (m, 1H); 4.60-4.10 (m, 5H); 3.58 (dd, 1H, J = 9.2, 4.8 Hz); 3.51 (dd, 1H, J = 9.2, 5.9 Hz); 3.00 (q, 1H, J = 7.4 Hz); 2.7 (ddt, 1H, J = 12.8, 8.8, 2.5 Hz); 2.35 (m, 1H); 2.15 (m, 1H); 1.75 (m, 1H). ¹³C NMR δ (ppm): 138.2; 128.4; 127.7; 83.8; 75.3; 73.4; 65.9; 49.7; 43.5; 40.4; 38.3; 32.6. *Minor isomer.* ¹H NMR δ (ppm): 7.40-7.20 (m, 5H); 4.60-4.10 (m, 7H); 3.75-3.65 (dd, 1H, J = 9.6, 3.6 Hz); 3.45-3.40 (m, 1H); 2.98-2.85 (m, 1H); 2.50-2.40 (m, 1H); 2.35-2.25 (m, 2H); 1.80-1.70 (m, 1H). ¹³C NMR δ (ppm): 138.2; 128.5; 127.7; 85.3; 73.3; 67.9; 66.3; 49.1; 42.4; 34.0; 32.1; 31.1. IR (film), ν_{max} (cm⁻¹): 3338, 2931, 2856, 1451, 1362, 1208, 1101, 1069, 733, 696. m/z: 374 (<1%); 356 (<1%); 155 (6%); 121 (8%); 91 (100%); 79 (13%); 65 (13%); 41 (8%). Anal calcd for C₁₅H₁₉IO₃: C, 48.14; H, 5.12. Found C, 48.21; H, 5.19.

Checking Results for CIF (ROSINI.CIF)

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The file contains the following data block(s)

"rosiniL"

PLATON results for rosini

TIME: Sep 16 17:42:20 2003

INDEX for rosini

- Crystal Data
 - Orthogonalization Matrices
 - Space Group Symmetry
 - ADDSYM
 - Coordinates
 - Anisotropic Displacement Parameters (U^{ij})
 - Rigid Body Analysis
 - Intramolecular Geometry
 - Bond Types and Chirality
 - Rings and Least-Squares Planes
 - Ring Puckering Analysis
 - Summary
-

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Crystal Data

Input Cell (Lattice Type: P)			-	Temp = 293K			Reduced Cell			(Acta Cryst. (1976), A32, 297-298)		
a =	8.4301 (6)	Angstrom			90	Degree	a =	8.430	alpha =	90.00	V =	2176.4
b =	11.7520 (9)				90		b =	11.752	beta =	90.00		
c =	21.9680 (17)				90		c =	21.968	gamma =	90.00		
V =	2176.4 (3)	Cubic-Angstrom			8.4301	Angstrom			Niggli Values			
					11.7520				71.067	138.110	482.593	
Lambda (MoKa) =	0.71073	Angstrom			21.9680				0.000	0.000	0.000	

Orthogonalization Matrices

(See e.g. J.D.Dunitz, Xray Analysis and Structure Determination of Organic Molecules, Cornell Univ. Press, 1979, P236)												
(XO) = (	8.43010	0	0)	(X) ,	(X) = (	0.11862	0	0)	(XO)	Orthogonal Axes AO, BO and CO		
(YO) = (	0	11.75200	0)	*(Y) ,	(Y) = (	0	0.08509	0)	*(YO)	are defined as:		
(ZO) = (	0	0	21.96800)	(Z) ,	(Z) = (	0	0	0.04552)	(ZO)	AO // A, CO // C*, BO // CO X AO		

Space Group Symmetry

(See e.g. G. Burns & A.M. Glazer, Space Groups for Solid State Scientists, Academic Press, 1990 or Int. Tables A)												
Space Group Pbca			No: 61, Laue: mmm [Hall: -P 2ac 2ab]									
Lattice Type oP, Centric, Orthorhombic, Order 8(4)			[Schoenflies: D2h ¹⁵]									
Nr	***** Symmetry Operation(s) *****											
1	X ,	Y ,	Z									
2	1/2 - X ,	- Y ,	1/2 + Z									
3	1/2 + X ,	1/2 - Y ,	- Z									

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4      - X ,      1/2 + Y ,      1/2 - Z
5      - X ,      - Y ,      - Z
6      1/2 + X ,      Y ,      1/2 - Z
7      1/2 - X ,      1/2 + Y ,      Z
8      X ,      1/2 - Y ,      1/2 + Z
```

ADDSYM

ADDSYM - CHECK (cf. MISSYM (C): Le Page, Y., *J. Appl. Cryst.* (1987), 20, 264-269; *J. Appl. Cryst.* (1988), 21, 983-984)

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- ADDSYM Search on ALL NON-H Chemical Types [Max NonFit 20 Perc]
- Number of Input Atoms Included in Search 12 (Unitcell 96)
- Density based on Input Atom Set = 1.722 g.cm-3 - Vol / Non-H atom = 22.7 Ang+3
- The Structure Implies the Following Symmetry Elements Subject to the Criteria:
  1.00 Deg (Metric), 0.25 Ang (Rotations), 0.45 Ang (Inv), 0.45 Ang (Transl)
Symm. Input Reduced (Ang) (Deg) (%) (Ang)
Elem Cell Row Cell Row d Typ Dot Angle Fit MaxDev. x y z
-----
b [ 1 0 0 ] [ 1 0 0 ] 8.43 2 1 0.00 100 0 through 1/4 0 0
   Glide = 0 1/2 0
c [ 0 1 0 ] [ 0 -1 0 ] 11.75 2 1 0.00 100 0 through 0 1/4 0
   Glide = 0 0 1/2
a [ 0 0 1 ] [ 0 0 1 ] 21.97 2 1 0.00 100 0 through 0 0 1/4
   Glide = 1/2 0 0
-1 ===== 100 0 at 0 0 0
   T.R.A.N.S.F.O.R.M.A.T.I.O.N M.A.T.R.I.X for CELL and HKL DATA
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Reduced->Convent      Input->Reduced      T = Input->Convent:      a' = T a
-----
( 1 0 0 ) ( 1 0 0 ) ( 1 0 0 ) Det(T)
( 0 1 0 ) X ( 0 -1 0 ) = ( 0 -1 0 ) =
( 0 0 1 ) ( 0 0 -1 ) ( 0 0 -1 ) 1.000
Cell Lattice a b c Alpha Beta Gamma Volume CrystalSystem Iaue
-----
Input oP 8.430 11.752 21.968 90.00 90.00 90.00 2176 Orthorhombic mmm
Reduced P 8.430 11.752 21.968 90.00 90.00 90.00 2176
Convent oP 8.430 11.752 21.968 90.00 90.00 90.00 2176 Orthorhombic mmm
:: *** No Obvious Extra Crystallographic Symmetry was Detected ***
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(1:19-Rule Rounded) Coordinates of Unique Residue(s) Identified. Standard Deviations in the Last Digit are in Parentheses.

 Site = Site Symmetry; SSN = Site Symmetry Number; SSOF = SHEILX Site Occupation Factor (= S.O.F / SSN).

 Site = Transformation on Input Data: N.IJK (N = SymOp, IJK = Translation) i.e. 1.555 = nomove
 SYMBOLS: Type = D/A = Potential Donor or Acceptor atom, D-H = H on Donor atom, MET = Metal.

 El Type = AK = Alkali Metal, AE = Alkaline Earth, HL = Halogen, AN = Actinide, LN = Lanthanide, TR = Transition Element.
 ARU = Asymmetric Residue Unit encoded as sklm.nn, with s = symmetry op, klm = translation, nn = residue #.
 Residue = collection of ARU's constituting an isolated unit (= molecule, ion).
 FLAGS : d = determined, c = calculated, R = riding G = group

Atom Types : C H I O
 Cov.Rad(Ang) : 0.68 0.35 1.40 0.68
 Atom Number : 6 1 53 8
 Atom Weight : 12.010 1.008 126.90 16.00
 Scat.Fact.f0: 5.999 1.000 53.001 7.999
 Scat.Fact.f1: 0.003 0.000 -0.474 0.011
 Scat.Fact.f2: 0.002 0.000 1.812 0.006
 Mu/Rho(MoKa): 0.58 0.37 36.68 1.22
 Elem. Type : -- -- HL --

 Sources - Cov. Radii : Manual Cambridge Cryst. Data Base
 - Atomic Wt. : IUPAC 1993
 - mu/rho : International Tables C, table 4.2.4.2, p193-199

NOMOVE

Flags Label	Fractional Coordinates (x,y,z)			Orthogonal Coordinates (XO,YO,ZO)			Site SSN*SSOF =	S.O.F	Move Type
d I (1)	0.34260 (4)	0.19987 (3)	0.37172 (2)	2.8882 (3)	2.3489 (4)	8.1660 (4)	1	1	-
d O (2)	0.6326 (3)	0.3190 (2)	0.27056 (12)	5.333 (3)	3.749 (2)	5.944 (3)	1	1	-
d O (3)	0.9167 (3)	0.2616 (2)	0.31446 (11)	7.728 (3)	3.074 (2)	6.908 (2)	1	1	-
d C (1)	0.5712 (5)	0.2805 (3)	0.37791 (14)	4.815 (4)	3.296 (4)	8.302 (3)	1	1	-
d C (4)	0.7529 (4)	0.4100 (3)	0.42570 (14)	6.347 (3)	4.818 (4)	9.352 (3)	1	1	-
d C (5)	0.6064 (4)	0.3706 (3)	0.32867 (14)	5.112 (3)	4.355 (4)	7.220 (3)	1	1	-
d C (6)	0.9147 (4)	0.3611 (3)	0.35169 (16)	7.711 (3)	4.244 (4)	7.726 (4)	1	1	-
d C (7)	0.7560 (4)	0.4274 (3)	0.35462 (15)	6.373 (3)	5.023 (4)	7.790 (3)	1	1	-
d C (8)	0.9041 (4)	0.3374 (3)	0.42006 (16)	7.622 (3)	3.965 (4)	9.228 (4)	1	1	-
d C (9)	0.4752 (5)	0.4575 (3)	0.3199 (2)	4.006 (4)	5.377 (4)	7.028 (4)	1	1	-
d C (10)	0.6023 (5)	0.3401 (3)	0.43822 (16)	5.077 (4)	3.997 (4)	9.627 (4)	1	1	-
d C (11)	0.7677 (7)	0.5149 (4)	0.46515 (19)	6.472 (6)	6.051 (5)	10.218 (4)	1	1	-
CR H (1)	0.65120	0.22060	0.37370	5.4897	2.5925	8.2094	1	1	-
CR H (2)	0.71660	0.28380	0.27110	6.0410	3.3352	5.9555	1	1	-
CR H (3)	0.98160	0.26980	0.28700	8.2750	3.1707	6.3048	1	1	-
CR H (6)	1.00370	0.41170	0.34210	8.4613	4.8383	7.5153	1	1	-
CR H (7)	0.76660	0.50730	0.34260	6.4625	5.9618	7.5262	1	1	-
CR H (8A)	0.99310	0.36740	0.44290	8.3719	4.3177	9.7296	1	1	-
CR H (8B)	0.88720	0.25770	0.42960	7.4792	3.0285	9.4375	1	1	-
CR H (9A)	0.50540	0.51040	0.28870	4.2606	5.9982	6.3422	1	1	-
CR H (9B)	0.45800	0.49780	0.35730	3.8610	5.8501	7.8492	1	1	-
CR H (9C)	0.37930	0.41940	0.30820	3.1975	4.9288	6.7705	1	1	-
CR H (10A)	0.61990	0.28540	0.47060	5.2258	3.3540	10.3381	1	1	-
CR H (10B)	0.51430	0.38910	0.44930	4.3356	4.5727	9.8702	1	1	-
CR H (11A)	0.86410	0.55430	0.45530	7.2844	6.5141	10.0020	1	1	-
CR H (11B)	0.76960	0.49290	0.50720	6.4878	5.7926	11.1422	1	1	-

cR H(11C) 0.67880 0.56420 0.45800 5.7224 6.6305 10.0613 "rosini " PLATON-GEOMETRY Page 4

Ordered Structure Unit Cell Contents (Based on Contents of Atom List, that may be Incomplete)

Resd Site X(cen) Y(cen) Z(cen) Res.Mol.Wt. Z C H I O

1 1 0.551 0.293 0.369 282.11 8 9 15 1 2

Unit Cell Weight = 2256.88

Calculated Analysis (Percent) = 72 120 8 16

Moiety Formula = C9 H15 I O2 38.3 5.4 45.0 11.3

Sum Formula = C9 H15 I O2

Formula Weight = 282.11

Formula_Z = 8

SpaceGroup_Z = 8 ==> Z' = 8 / 8 = 1.000

Calculated Density = 1.7220(2) g cm-3 [= Mg m-3]

F(000) = 1104.0 [1100.64]

mu (MoKa) = 29.07 cm-1 = 2.907 mm-1

Note on F000: The first number is a pure electron count, the second number between [] is calculated from f,f' & f"

"rosini " PLATON-MOLSYM

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MOLSYM: Search for (additional) Molecular (Point Group) Symmetry - Hydrogen Atoms Excluded ! - Ordered Residues Only !

For Details on the Molecular Symmetry Determination see: T. Pilati & A. Forni, J.Appl.Cryst. (1998), 31, 503-504 & (2000), 33, 417.

For CSM (i.e. Continuous Symmetry Measure), see: H. Zabrodsky et al. (1993) JACS, 115, 8278-8289

:: Resd # 1, No Molecular Symmetry Within Tolerance = 0.80 Ang.

"rosini " PLATON-NONSYM

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NONSYM Search for Additional (Non)Crystallographic Symmetry between Residues (Experimental)

- Residue numbers with opposite signs indicate potential enantiomeric pairs

- Hydrogen atoms omitted from the analysis

- Residues with more than 6 atoms are analysed only

RES# Coords Center of Gravity Main axes (hor) EigenV Asym Angle a,b,c

-1 0.5413 0.2872 0.3684 0.425 -0.830 0.363 1348 79.78 65 146 69

4.5636 3.3753 8.0922 0.196 -0.307 -0.931 1234 20.41 79 108 159

Res.Mol.Wt. = 266.99 -0.884 -0.467 -0.032 394 188.23 152 118 92

"rosini " PLATON-ADP-Anal

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(An)isotropic, Equivalent and Main Axes Displacement Parameters - Unusual Values Marked with a # - [Optional Coordinate Split-up]

Atom	Label	U11 or Uiso	U22	U33	U23	U13	U12	Ueq(sUeq)	U1	U2	U3	U3/U1
1	I1	0.0455(2) [0.3330	0.1003(3) 0.2126	0.0942(4) 0.3746]	0.02777(18) [0.3522	-0.00774(12) 0.1872	-0.02771(14) 0.3688]	0.08000(18)	0.0337	0.0733	0.1330	3.95
2	O2	0.0417(13) [0.6327	0.0772(16) 0.3267	0.0378(13) 0.2704]	-0.0035(10) [0.6325	-0.0039(10) 0.3113	-0.0001(12) 0.2708]	0.0522(8)	0.0352	0.0440	0.0775	2.20
3	O3	0.0401(13) [0.9130	0.0637(13) 0.2675	0.0520(14) 0.3134]	-0.0075(12) [0.9204	0.0109(10) 0.2557	-0.0024(11) 0.3155]	0.0519(8)	0.0335	0.0530	0.0693	2.07
4	C1	0.0334(16) [0.5703	0.0490(14) 0.2847	0.0461(18) 0.3789]	0.0072(13) [0.5721	0.0004(12) 0.2763	-0.0027(13) 0.3770]	0.0428(9)	0.0327	0.0407	0.0551	1.68
5	C4	0.0497(17) [0.7579	0.0441(14) 0.4103	0.0328(14) 0.4257]	0.0011(11) [0.7479	0.0010(13) 0.4097	0.0007(13) 0.4257]	0.0422(9)	0.0326	0.0441	0.0499	1.53
6	C5	0.0333(14) [0.6082	0.0506(15) 0.3746	0.0352(15) 0.3290]	0.0046(12) [0.6046	0.0017(12) 0.3666	0.0039(12) 0.3283]	0.0397(8)	0.0322	0.0341	0.0528	1.64
7	C6	0.0335(15) [0.9108	0.0546(17) 0.3664	0.0416(16) 0.3515]	-0.0001(13) [0.9186	0.0043(13) 0.3558	-0.0089(12) 0.3519]	0.0432(9)	0.0290	0.0427	0.0580	2.00
8	C7	0.0406(16) [0.7532	0.0451(14) 0.4301	0.0344(14) 0.3549]	0.0045(12) [0.7588	-0.0016(12) 0.4247	-0.0037(12) 0.3543]	0.0400(8)	0.0328	0.0386	0.0487	1.49
9	C8	0.0446(18) [0.9045	0.0597(17) 0.3424	0.0423(18) 0.4204]	0.0044(14) [0.9037	-0.0072(14) 0.3324	0.0023(15) 0.4197]	0.0489(10)	0.0352	0.0506	0.0608	1.73
10	C9	0.051(2) [0.4772	0.062(2) 0.4641	0.069(2) 0.3221]	0.0117(18) [0.4732	0.0092(18) 0.4509	0.0148(16) 0.3177]	0.0607(12)	0.0347	0.0694	0.0779	2.24
11	C10	0.0477(18) [0.6044	0.0610(18) 0.3460	0.0391(17) 0.4389]	0.0087(14) [0.6002	0.0076(14) 0.3342	0.0000(15) 0.4375]	0.0493(10)	0.0326	0.0507	0.0645	1.98
12	C11	0.094(3) [0.7806	0.062(2) 0.5133	0.046(2) 0.4653]	-0.0119(17) [0.7548	0.001(2) 0.5165	-0.007(2) 0.4650]	0.0673(14)	0.0396	0.0667	0.0957	2.42
13	H1	0.05100						0.05100				
14	H2	0.07800						0.07800				
15	H3	0.07800						0.07800				
16	H6	0.05200						0.05200				
17	H7	0.04800						0.04800				
18	H8A	0.05900						0.05900				
19	H8B	0.05900						0.05900				
20	H9A	0.09100						0.09100				
21	H9B	0.09100						0.09100				
22	H9C	0.09100						0.09100				
23	H10A	0.05900						0.05900				
24	H10B	0.05900						0.05900				
25	H11A	0.10100						0.10100				
26	H11B	0.10100						0.10100				
27	H11C	0.10100						0.10100				
U(i,j)-Average		0.0463	0.0608	0.0475	0.0039	-0.0002	-0.0026	"rosini	0.0456	0.0467	0.0622	1.36
									" PLATON-ADP-Anal	Page	8	

The Displacement Factor has the Form of Exp(-T)

where

$$T = 2 * (\pi * 2) * (U11 * (h * as) ** 2 + U22 * (k * bs) ** 2 + U33 * (l * cs) ** 2 + 2 * U23 * k * l * bs * cs + 2 * U13 * h * l * as * cs + 2 * U12 * h * k * as * bs), \text{ for Anisotr. Atoms}$$
$$U_{eq} = 1/3 \sum(i, j) (U_{ij} * as(i) * as(j) * a(i) * a(j))$$

U1, U2, U3 are the three Main Axes Components of Uij

Reference U(eq): R.X. Fischer & E. Tillmanns, Acta Cryst. (1988). C44, 775-776

Ueq [or U(iso)] Averages per Element

	Non-H	C	H	I	O
Average	0.0515	0.0482	0.0746	0.0800	0.0521
Minimum	0.0397	0.0397	0.0480	0.0800	0.0519
Maximum	0.0800	0.0673	0.1010	0.0800	0.0522
Ratio	2.0151	1.6952	2.1042	1.0000	1.0058
Number	12	9	15	1	2

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V.Schomaker and K.N.Trueblood Rigid Body Motion Analysis, TLS - Model (Acta Cryst. (1968), B24, 63-76) - see also Dunitz, p244

Vibration Tensor in Inertial System I(1)=L,I(2)=M, I(3)=N (Difference U(calc)-U(obs) in Parentheses)

Label	U(L,L)	U(L,M)	U(L,N)	U(M,M)	U(M,N)	U(N,N)	Ueq(obs)	Ueq(calc)
I(1)	0.03502(-.00035)	-0.01036(-.00242)	0.00446(0.00184)	0.11493(-.00300)	-0.02649(0.00711)	0.09005(0.00074)	0.0800	0.0791
O(2)	0.04902(-.00130)	-0.00020(0.00469)	0.01554(-.00860)	0.04109(0.00223)	-0.00933(-.00272)	0.06659(-.00214)	0.0522	0.0511
O(3)	0.04367(0.00584)	-0.00887(0.00309)	0.00514(0.00785)	0.04467(0.00091)	0.00581(0.00345)	0.06746(-.00031)	0.0519	0.0541
C(1)	0.03482(-.00015)	-0.00566(-.00180)	0.00329(-.00043)	0.05018(0.00103)	-0.00510(-.00145)	0.04350(0.00062)	0.0428	0.0433
C(4)	0.04913(-.00060)	-0.00012(-.00020)	-0.00209(-.00021)	0.03469(0.00010)	-0.00448(-.00130)	0.04278(0.00431)	0.0422	0.0435
C(5)	0.04052(-.00071)	-0.00667(0.00302)	0.00754(-.00229)	0.03811(0.00492)	-0.00511(-.00227)	0.04046(-.00001)	0.0397	0.0411
C(6)	0.03101(0.00293)	-0.00513(0.00072)	0.00189(0.00288)	0.04195(0.00099)	-0.00378(-.00115)	0.05674(-.00166)	0.0432	0.0441
C(7)	0.03857(0.00072)	-0.00049(-.00165)	-0.00027(-.00254)	0.03925(-.00189)	-0.00782(0.00146)	0.04229(-.00169)	0.0400	0.0391
C(8)	0.04951(-.00075)	0.00142(0.00102)	0.00888(-.00399)	0.04889(0.00047)	-0.00919(-.00125)	0.04820(0.00018)	0.0489	0.0458
C(9)	0.06545(0.00034)	-0.00246(-.00514)	0.01337(0.00124)	0.07591(-.00294)	-0.00311(-.00550)	0.04063(0.00139)	0.0607	0.0603
C(10)	0.05129(0.00029)	-0.01172(-.00249)	0.00147(0.00076)	0.04369(-.00139)	-0.00958(0.00085)	0.05283(-.00032)	0.0493	0.0488
C(11)	0.08090(0.00274)	0.01176(0.00116)	-0.01421(0.00349)	0.04303(-.00142)	-0.00192(0.00277)	0.07807(-.00112)	0.0673	0.0671
R1	Sum(abs(U(obs)-U(calc)))/Sum(abs(U(obs))) = 0.07479							
R2	Sqrt(Sum((U(obs)-U(calc))**2)/Sum(U(obs)**2)) = 0.07646							
S	Sqrt(Sum((U(obs)-U(calc))**2)/(6*N-NS*M)) = 0.00351							
N	Number of Atoms in Rigid Group = 12							
NS	Symmetry Factor = 1							
M	Number of Rigid-Body Parameters = 20							
Largest abs(U(obs)-U(calc)) = 0.00975								TLS-Mode
								"rosini"
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Inertial Tensor I, Eigenvectors and Eigenvalues of I in the Cartesian XO,YO,ZO System and Angular Relation with X,Y,Z System

XO	YO	ZO	Value	X	Y	Z	Origin (Mass-Weighted)
I(1)	0.88381	0.46673	0.03236	1348.43	27.90	62.18	88.15 X = 0.54135
I(2)	0.19607	-0.30670	-0.93139	1234.13	78.69	107.86	158.65 Y = 0.28721
I(3)	-0.42479	0.82952	-0.36257	394.16	115.14	33.95	111.26 Z = 0.36836
Librational Tensor, L(rad**2) Eigenvalues and Eigenvalues of L in the Inertial System XI,YI,ZI							
				XI	YI	ZI	rad**2 deg**2
0.00614(.00050)-0.00059(.00035)-0.00037(.00035) 0.00462(.00034)0.00127(.00029)0.00494(.00030)				L(1)	0.73260	-0.48183	-0.48077 0.00678 22.25
				L(2)	0.67609	0.43346	0.59583 0.00543 17.84
				L(3)	-0.07869	-0.76155	0.64331 0.00349 11.44
Translational Tensor, T(ang**2) Eigenvalues and Eigenvalues of T in the Inertial System XI,YI,ZI							
				XI	YI	ZI	Ang^2
0.03484(.00116)-0.00756(.00119)0.00468(.00130) 0.05376(.00153)-0.00680(.00144)0.04639(.00158)				T(1)	-0.32352	0.81175	-0.48621 0.06085
				T(2)	0.01185	0.51728	0.85574 0.04234
				T(3)	0.94615	0.27108	-0.17697 0.03180
Cross Tensor, S(rad*Ang)							
0.00085(.00047)0.00139(.00080)-0.00039(.00078) 0.00204(.00047)-0.00227(.00048)0.00675(.00047) 0.00233(.00036)-0.00380(.00045)0.00142(.00067)				Calculation of the Origin Shift that Symmetrizes S			
Shift Origin in I				New S-Tensor	New T-Tensor	Eigenvalues and Values of New-T in I-System	
Rol(1) 1.24764				0.00096	0.00138	0.00150	0.03394 -0.00325 0.00152 New T(1) -0.39902 0.60521 -0.68884 0.04149
Rol(2) 1.16681				0.00138	-0.00014	-0.00131	-0.00325 0.03486 -0.00394 New T(2) -0.70352 0.27974 0.65330 0.03383
Rol(3) -0.31050				0.00150	-0.00131	-0.00082	0.00152 -0.00394 0.03715 New T(3) 0.58808 0.74530 0.31416 0.03063
Angular Relationships (Degrees)							
Libration Axes -				Inertial Axes	Libration Axes - Crystal Axes		
				XI	YI	ZI	X Y Z
L(1)				42.90	118.80	118.74	40.78 84.78 49.70
L(2)				47.46	64.31	53.43	64.57 47.40 126.72
L(3)				94.51	139.60	49.96	119.48 43.07 61.74
Translation Axes -				Inertial Axes	Translation Axes - Crystal Axes		
				XI	YI	ZI	X Y Z
New T(1)				113.52	52.76	133.54	86.64 160.61 109.08
New T(2)				134.71	73.76	49.21	147.61 82.66 121.34
New T(3)				53.98	41.82	71.69	57.83 72.15 142.10

Bond		Bond Distance		Components of the Correction			Vibration Along the Interatomic Bond			Angle with Lib. Axes			
Atom(I)	Atom(J)	Obsd	Calcd	Del(L)	Del(M)	Del(N)	I to J	J to I	Difference	Sqrt(Diff)	L(1)	L(2)	L(3)
I (1)	- C (1)	2.1518	2.1620	-0.0102	-0.0005	0	0.0351(3)	0.0348(16)	0.0003(16)	0.0185	139.38	130.16	84.88
O (2)	- C (5)	1.4304	1.4384	-0.0002	0.0079	-0.0016	0.0434(14)	0.0404(15)	0.0030(20)	0.0547	119.92	71.76	143.92
O (3)	- C (6)	1.4271	1.4353	-0.0025	0.0065	-0.0045	0.0527(13)	0.0503(16)	0.0024(21)	0.0486	115.86	101.94	151.13
C (1)	- C (5)	1.5425	1.5504	-0.0039	-0.0036	-0.0058	0.0392(16)	0.0385(15)	0.0007(22)	0.0267	75.79	165.13	94.28
C (1)	- C (10)	1.5214	1.5293	-0.0025	0.0076	-0.0009	0.0518(16)	0.0533(18)	0.0015(24)	0.0381	137.36	82.24	131.59
C (4)	- C (7)	1.5750	1.5829	-0.0009	-0.0073	-0.0030	0.0327(15)	0.0335(15)	0.0008(21)	0.0279	51.91	133.45	67.50
C (4)	- C (8)	1.5388	1.5475	-0.0034	-0.0044	0.0067	0.0472(15)	0.0485(18)	0.0013(23)	0.0357	121.65	91.64	31.70
C (4)	- C (10)	1.5370	1.5445	0.0073	0.0017	0.0014	0.0477(15)	0.0473(18)	0.0004(23)	0.0189	56.06	34.57	95.79
C (4)	- C (11)	1.5121	1.5206	-0.0032	0.0066	-0.0044	0.0416(15)	0.0450(23)	0.0034(28)	0.0579	120.48	104.16	145.72
C (5)	- C (7)	1.5366	1.5436	-0.0067	0.0022	0.0004	0.0421(15)	0.0384(15)	0.0037(21)	0.0608	154.28	115.13	95.11
C (5)	- C (9)	1.5177	1.5261	0.0023	0.0030	-0.0076	0.0369(15)	0.0381(20)	0.0012(25)	0.0345	55.06	102.62	142.19
C (6)	- C (7)	1.5495	1.5581	0.0038	0.0041	-0.0066	0.0463(16)	0.0452(15)	0.0010(22)	0.0320	54.46	86.83	144.28
C (6)	- C (8)	1.5302	1.5379	0.0013	0.0070	0.0032	0.0414(16)	0.0422(18)	0.0008(24)	0.0288	125.04	42.68	111.13
Sqrt(Sum(DelIJ**2)/Nrbd) =										0.0019			

Sqrt(Sum(Del(IJ**2)/Nrb) = 0.0019

- Indicates bonds exceeding the 5.0 sigma test level
Test Matrix for Rigid-Body Vibrations - /Del(A,B)/ = /Z(A,B)**2 - Z(B,A)**2/ Should be Near Zero (Acta Cryst A34,1978,828)

Atom-Atom	1	2	3	4	5	6	7	8	9	10	11	12	
1 I(1)	-	0	0	8	0	1	2	4	1	8	2	1	1
2 O(2)	-	4	0	11	1	1	-3	2	5	2	3	1	1
3 O(3)	-	5	3	0	2	3	10	-2	1	0	6	5	2
4 C(1)	-	-2	2	3	0	2	-1	4	1	7	0	-1	2
5 C(4)	-	4	4	3	2	0	0	1	-1	-1	3	0	-3
6 C(5)	-	3	-1	3	-2	3	0	3	-4	1	-1	3	1
7 C(6)	-	5	3	-1	3	2	3	0	-1	-1	2	6	5
8 C(7)	-	4	2	3	2	-2	-2	0	1	3	1	1	1
9 C(8)	-	5	4	2	3	-2	3	-2	2	0	1	2	6
Remarks													

-	Upper Triangle Entries Represent /Del (A,B)/*1000 Values												
-	Lower Triangle Entries Represent Distances (A-B) Angstrom												
-	Negative Entries Indicate Bonded Atoms												

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- Upper Triangle Entries Represent /Del(A,B)/*1000 Values
- Lower Triangle Entries Represent Distances (A-B) Angstrom
- Negative Entries Indicate Bonded Atoms

Bond Types and Chirality

Analysis of Bond Distance and Angle Values - Identification of Chiral Center(s) and Their (R/S)-Configuration (Cahn-Ingold-Prelog)

The Following Tests are done. Faults are Marked Under Note
-- V : Valency Check Fault for H, C
-- S : Bond Too Short
-- A : Unusual Bond Angle Values (PLEASE CHECK)

*** PLEASE NOTE: R/S ASSIGNMENTS ARE TENTATIVE *** (CIP Special rules NOT Implemented)																				
*** See Angew.Chem.Intern. Ed. Eng., (1966),5,385 & (1982),21,567 for Authoritative Details for Special Cases																				
=A.N.G.L.E.S.=B.O.N.D.S=																				
Flag Label	- Connected to				(May be Incomplete for Polymeric Structures)				nra	min	max	Aver	min	max	nrb	tnr	Hyb	RS	Note	
d I(1)	-	C(1)	-----	-----	-----	-----	-----	-----	0	0	0	0.0	2.152	2.152	1	21				
d O(2)	-	C(5)	-----	-----	-----	-----	-----	-----	1	110	110	109.5	0.820	1.430	2	20	sp3			
d O(3)	-	C(6)	-----	-----	-----	-----	-----	-----	1	109	109	109.5	0.820	1.427	2	19	sp3			
d C(1)	-	I(1)	-----	-----	-----	-----	-----	-----	6	105	115	109.4	0.979	2.152	4	18	sp3	R		
d C(4)	-	C(7)	-----	-----	-----	-----	-----	-----	6	89	117	109.0	1.512	1.575	4	11	sp3	R		
d C(5)	-	O(2)	-----	-----	-----	-----	-----	-----	6	101	114	109.5	1.430	1.543	4	17	sp3	R		
d C(6)	-	O(3)	-----	-----	-----	-----	-----	-----	6	90	116	109.2	0.980	1.550	4	16	sp3	R		
d C(7)	-	C(4)	-----	-----	-----	-----	-----	-----	6	89	118	109.1	0.979	1.575	4	14	sp3	S		
d C(8)	-	C(4)	-----	-----	-----	-----	-----	-----	6	92	113	109.3	0.969	1.539	4	12	sp3			
d C(9)	-	C(5)	-----	-----	-----	-----	-----	-----	6	109	110	109.5	0.959	1.518	4	13	sp3			
d C(10)	-	C(1)	-----	-----	-----	-----	-----	-----	6	103	111	109.5	0.970	1.537	4	15	sp3			
d C(11)	-	C(4)	-----	-----	-----	-----	-----	-----	6	109	110	109.5	0.959	1.512	4	10	sp3			
cR H(1)	-	C(1)	-----	-----	-----	-----	-----	-----	0	0	0	0.0	0.979	0.979	1	7				
cR H(2)	-	O(2)	-----	-----	-----	-----	-----	-----	0	0	0	0.0	0.820	0.820	1	9				
cR H(3)	-	O(3)	-----	-----	-----	-----	-----	-----	0	0	0	0.0	0.820	0.820	1	8				
cR H(6)	-----	C(6)	-----	-----	-----	-----	-----	-----	0	0	0	0.0	0.980	0.980	1	6				
cR H(7)	-----	C(7)	-----	-----	-----	-----	-----	-----	0	0	0	0.0	0.979	0.979	1	4				
cR H(8A)	-----	C(8)	-----	-----	-----	-----	-----	-----	0	0	0	0.0	0.969	0.969	1	2				
cR H(8B)	-----	C(8)	-----	-----	-----	-----	-----	-----	0	0	0	0.0	0.970	0.970	1	2				
cR H(9A)	-----	C(9)	-----	-----	-----	-----	-----	-----	0	0	0	0.0	0.960	0.960	1	3				
cR H(9B)	-----	C(9)	-----	-----	-----	-----	-----	-----	0	0	0	0.0	0.959	0.959	1	3				
cR H(9C)	-----	C(9)	-----	-----	-----	-----	-----	-----	0	0	0	0.0	0.959	0.959	1	3				
cR H(10A)	-----	C(10)	-----	-----	-----	-----	-----	-----	0	0	0	0.0	0.970	0.970	1	5				
cR H(10B)	-----	C(10)	-----	-----	-----	-----	-----	-----	0	0	0	0.0	0.970	0.970	1	5				
cR H(11A)	-----	C(11)	-----	-----	-----	-----	-----	-----	0	0	0	0.0	0.960	0.960	1	1				
cR H(11B)	-----	C(11)	-----	-----	-----	-----	-----	-----	0	0	0	0.0	0.959	0.959	1	1				
cR H(11C)	-----	C(11)	-----	-----	-----	-----	-----	-----	0	0	0	0.0	0.960	0.960	1	1				
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Intramolecular Geometry

Analysis of the Intramolecular Geometry in Terms of Unique Molecule(s)/Ions, with Bond Criterion: $d(i-j) < R(i) + R(j) + Tol$

 -- Tol = 0.40 Ang. for Normal Bonds + 0.70 for (Earth)alkali-NonMetal Contacts and adjusted by -0.40 Ang. for Metal-Metal Distances
 -- The Bond Distance and Angle esd's have been Incremented to Include the Effect of the Unit-cell esd.
 (Rel.Error in Dist. 0.0001 Ang. , Abs. Angle Error 0.000 Deg.)

Bond Lengths (Angstrom). - (Bonds are ordered on the first label, left to right and top to bottom) - esd in last digit in ().

I (1)	-	C (1)	2.152(4)	O (2)	-	C (5)	1.430(4)	O (3)	-	C (6)	1.427(4)	C (1)	-	C (5)	1.543(5)
C (1)	-	C (10)	1.521(5)	C (4)	-	C (7)	1.575(5)	C (4)	-	C (8)	1.539(5)	C (4)	-	C (10)	1.537(5)
C (4)	-	C (11)	1.512(6)	C (5)	-	C (7)	1.537(5)	C (5)	-	C (9)	1.518(5)	C (6)	-	C (7)	1.550(5)
C (6)	-	C (8)	1.530(5)												
O (2)	-	H (2)	0.8202	O (3)	-	H (3)	0.8201	C (1)	-	H (1)	0.9792	C (6)	-	H (6)	0.9803
C (7)	-	H (7)	0.9795	C (8)	-	H (8A)	0.9690	C (8)	-	H (8B)	0.9703	C (9)	-	H (9A)	0.9597
C (9)	-	H (9B)	0.9594	C (9)	-	H (9C)	0.9592	C (10)	-	H (10A)	0.9702	C (10)	-	H (10B)	0.9701
C (11)	-	H (11A)	0.9600	C (11)	-	H (11B)	0.9594	C (11)	-	H (11C)	0.9602				

Bond/Valence Angles (Degrees) - (Angles are ordered on the middle label, left to right and top to bottom) - esd in last digit in ().

I (1)	-	C (1)	-	C (5)	115.5(2)	I (1)	-	C (1)	-	C (10)	114.3(3)	C (5)	-	C (1)	-	C (10)	105.2(3)
C (7)	-	C (4)	-	C (8)	88.8(2)	C (7)	-	C (4)	-	C (10)	105.1(3)	C (7)	-	C (4)	-	C (11)	117.5(3)
C (8)	-	C (4)	-	C (10)	113.7(3)	C (8)	-	C (4)	-	C (11)	115.5(3)	C (10)	-	C (4)	-	C (11)	113.7(3)
O (2)	-	C (5)	-	C (1)	111.4(3)	O (2)	-	C (5)	-	C (7)	112.9(3)	O (2)	-	C (5)	-	C (9)	106.5(3)
C (1)	-	C (5)	-	C (7)	101.3(3)	C (1)	-	C (5)	-	C (9)	114.3(3)	C (7)	-	C (5)	-	C (9)	110.7(3)
O (3)	-	C (6)	-	C (7)	116.5(3)	O (3)	-	C (6)	-	C (8)	114.5(3)	C (7)	-	C (6)	-	C (8)	90.0(3)
C (4)	-	C (7)	-	C (5)	107.3(3)	C (4)	-	C (7)	-	C (6)	89.4(3)	C (5)	-	C (7)	-	C (6)	118.4(3)
C (4)	-	C (8)	-	C (6)	91.5(3)	C (1)	-	C (10)	-	C (4)	103.5(3)						
C (5)	-	O (2)	-	H (2)	109.52	C (6)	-	O (3)	-	H (3)	109.46	I (1)	-	C (1)	-	H (1)	107.11
C (5)	-	C (1)	-	H (1)	107.14	C (10)	-	C (1)	-	H (1)	107.13	O (3)	-	C (6)	-	H (6)	111.40
C (7)	-	C (6)	-	H (6)	111.39	C (8)	-	C (6)	-	H (6)	111.47	C (4)	-	C (7)	-	H (7)	113.16
C (5)	-	C (7)	-	H (7)	113.04	C (6)	-	C (7)	-	H (7)	113.08	C (4)	-	C (8)	-	H (8A)	113.45
C (4)	-	C (8)	-	H (8B)	113.34	C (6)	-	C (8)	-	H (8A)	113.38	C (6)	-	C (8)	-	H (8B)	113.35
H (8A)	-	C (8)	-	H (8B)	110.67	C (5)	-	C (9)	-	H (9A)	109.46	C (5)	-	C (9)	-	H (9B)	109.49
C (5)	-	C (9)	-	H (9C)	109.52	H (9A)	-	C (9)	-	H (9B)	109.39	H (9A)	-	C (9)	-	H (9C)	109.55
H (9B)	-	C (9)	-	H (9C)	109.42	C (1)	-	C (10)	-	H (10A)	111.09	C (1)	-	C (10)	-	H (10B)	111.10
C (4)	-	C (10)	-	H (10A)	111.04	C (4)	-	C (10)	-	H (10B)	111.06	H (10A)	-	C (10)	-	H (10B)	109.04
C (4)	-	C (11)	-	H (11A)	109.50	C (4)	-	C (11)	-	H (11B)	109.48	C (4)	-	C (11)	-	H (11C)	109.50
H (11A)	-	C (11)	-	H (11B)	109.44	H (11A)	-	C (11)	-	H (11C)	109.44	H (11B)	-	C (11)	-	H (11C)	109.46

Torsion/Dihedral Angles (Deg.) - Klyne & Prelog Convention (Dunitz, p241) - (Excl. Minor Disorder & Embedded Bond Angl. > 160. Deg.)

I (1)	C (1)	C (5)	O (2)	-71.9(3)	I (1)	C (1)	C (5)	C (7)	167.9(2)	I (1)	C (1)	C (5)	C (9)	48.9(4)
C (10)	C (1)	C (5)	O (2)	161.1(3)	C (10)	C (1)	C (5)	C (7)	40.9(4)	C (10)	C (1)	C (5)	C (9)	-78.1(4)
I (1)	C (1)	C (10)	C (4)	-168.4(2)	C (5)	C (1)	C (10)	C (4)	-40.7(4)	C (8)	C (4)	C (7)	C (5)	115.8(3)

[illegible][illegible]

Statistics of Bond Length per Bond Type (NOTE: A Indicates 10 Occurrences, B Indicates 11, Etc. and * more than 35)

Bond Type	Nr	B.P.	d(min)	d(max)	d(av)	Sumrad Note	0.0	0.5	1.0	1.5	2.0	2.5	3.0 Angstrom
I -- C	1	0.0040	2.1520	2.1520	2.1520	2.08							
O -- C ₄	2	0.0040	1.4270	1.4300	1.4285	1.36							
C ₄ -- C ₁₀	4	0.0051	1.5120	1.5750	1.5362	1.36							
O -- H	2	0	0.9201	0.8202	0.8201	1.03							
C -- H	13		0.9592	0.9803	0.9674	1.03							
Selected Bond Lengths (Angstrom) - see M.R.C. Ladd & R.A. Palmer, Structure Determination by X-Ray Crystallography (1985)													

Formal single bonds

C4-C4 1.54	C4-C3 1.52	C4-C2 1.46	C4-N3 1.47	C4-N2 1.47	C4-O2 1.43	C3-C3 1.46	C3-C2 1.45	C3-N3 1.40	C3-N2 1.40
C3-O2 1.36	C2-C2 1.38	C2-N3 1.33	C2-N2 1.33	C2-O2 1.36	N3-N3 1.45	N3-N2 1.45	N3-O2 1.36	N2-N2 1.45	N2-O2 1.41
Formal double bonds									

C3-C3 1.34	C3-C2 1.31	C3-N2 1.32	C3-O1 1.22	C2-C2 1.28	C2-N2 1.32	C2-O1 1.16	N3-O1 1.24	N2-N2 1.25	N2-O1 1.21
Formal triple bonds									

C2-C2 1.20	C2-N1 1.16	C3-C3 1.40	C2-N2 1.34	N2-N2 1.35					
The notation in the table indicates the connectivity of the atoms									
For more detailed standard bond distance tabulations see: J. Chem. Soc. Perkin II, (1987), S1-S19;									
J. Chem. Soc. Dalton Trans. (1989), S1 - S83 or International Tables C, (1992), 707-791.									
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Rings and Least-Squares Plane

Automatic Search for Rings (3 to 24-Membered) and Planes Determined by 4 or More Connected Atoms (with Deviation < 0.10 Ang.)

Least-Squares Planes - P*X+Q*Y+R*Z=S :: First Line Orthogonal(XO,YO,ZO), Second Line Fractional(X,Y,Z)

Ring/Plan/Resd/Lspl N Indicates that the Ring/Plane/Residue Involves N Atoms

Sigref - R.M.S-Error of the Contributing Atoms

Sigpln - Sqrt(Sum(j=1:N) (D(j)**2/(N-3))

Chisq - Chi-Squared = Sum(j=1:N) (D(j)**2)/Sigref**2

Pl.Hyp. - Result of the Chi.Sq. Test for Planarity (See Stout & Jensen, p424)

**** - Atoms Deviating by More Than 1.5 Angstrom and Hydrogen Atoms are NOT Listed

Note - Weights : UNIT

- Deviations from planes are in Angstrom Units

- **The Plane determining Atoms have been Marked #**

Nr	1	P	Q	R	S	Sigref	0.003	Sigpln	0.052	Chisq	232.8	Pl.Hyp.	P<5
Ring	0.5315(19)	0.8335(13)	0.151(2)	8.776(18)	#C(4)	0.026(3)	#C(6)	0.026(3)	#C(7)	-0.026(3)	#C(8)	-0.027(3)	
A 4	4.481(16)	9.795(15)	3.32(5)	8.776(18)	O(3)	-1.063(2)	C(5)	-1.338(3)	C(9)	-1.104(4)	C(10)	-1.292(4)	
C(11) 1.250(5)													
Nr	2	P	Q	R	S	Sigref	0.003	Sigpln	0.290	Chisq	13776.3	Pl.Hyp.	P<5
Ring	-0.6678(14)	0.7380(13)	0.0973(18)	0.29(2)	#C(1)	-0.267(4)	#C(4)	-0.065(3)	#C(5)	0.211(3)	#C(7)	-0.083(3)	
A 5	-5.630(12)	8.673(15)	2.14(4)	0.29(2)	#C(10)	0.204(4)	I(1)	0.307(1)	O(2)	-0.508(2)	C(11)	0.846(5)	
Nr	3	P	Q	R	S	Sigref	0.003	Sigpln	0.645	Chisq	136863.5	Pl.Hyp.	P<5
Ring	-0.1837(15)	0.9720(5)	0.147(2)	4.24(2)	#C(1)	-0.698(4)	#C(4)	0.654(4)	#C(5)	0.118(4)	#C(6)	-0.394(4)	
A 7	-1.548(13)	11.423(6)	3.23(5)	4.24(2)	#C(7)	0.619(4)	#C(8)	-0.428(4)	#C(10)	0.129(4)	I(1)	-1.285(1)	

O(2) -0.700(2) C(9) 1.285(4)

Nr	4	P	Q	R	S	Sigref	0.003	Sigpln	1.004	Chisq	784177.0	Pl.Hyp.	P<5
Resd	-0.1415(12)	0.9025(7)	-0.4068(18)	-0.342(15)	#I(1)	-1.269(1)	#O(2)	0.553(2)	#O(3)	-0.787(2)	#C(1)	-0.742(3)	
A 12	-1.193(10)	10.606(9)	-8.94(4)	-0.342(15)	#C(4)	-0.012(3)	#C(5)	0.612(3)	#C(6)	-0.062(4)	#C(7)	0.804(3)	
					#C(8)	-0.912(4)	#C(9)	1.769(4)	#C(10)	-0.686(4)	#C(11)	0.731(5)	

Nr	5	P	Q	R	S	Sigref	0.003	Sigpln	0.018	Chisq	25.9	Pl.Hyp.	P<5
Plan		-0.514(2)	0.8503(13)	0.1144(17)	1.90(2)	#C(4)	0.009(3)	#C(5)	0.006(3)	#C(7)	-0.009(3)	#C(10)	-0.006(4)
A 4		-4.330(17)	9.993(15)	2.51(4)	1.90(2)	I(1)	-0.450(1)	O(2)	-0.769(2)	C(1)	-0.618(4)	C(6)	-1.366(3)
						C(8)	-1.385(3)	C(9)	1.420(4)	C(11)	1.092(5)		

(Acute) Angles (Degrees) Between Planes (Numbers I,J from List Above)

1, 2= 74.1(2) 1, 3= 42.7(2) 1, 4= 52.0(2) 1, 5= 63.1(2) 2, 3= 31.3(2) 2, 4= 43.9(2) 2, 5= 11.0(2)
3, 4= 32.5(2) 3, 5= 20.4(2) 4, 5= 37.5(2)

(Acute) Angles (Degrees) Between Axes, Lines, Bonds and L.S.-Plane

Bond	/	Plane	NM	--> M	1	2	3	4	5	6	7	8	9	10

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Axis	O	----	a	/	OM	=	32.11(18),	41.90(15),	10.58(17),	8.13(13),	30.91(17),
	b	/	OM	=	56.46(18),	47.56(15),	76.40(17),	64.49(13),	58.25(17),		
	c	/	OM	=	8.68(18),	5.58(15),	8.44(17),	24.01(13),	6.57(17),		
	a*	/	OM	=	32.11(18),	41.90(15),	10.58(17),	8.13(13),	30.91(17),		
	b*	/	OM	=	56.46(18),	47.56(15),	76.40(17),	64.49(13),	58.25(17),		
	c*	/	OM	=	8.68(18),	5.58(15),	8.44(17),	24.01(13),	6.57(17),		
	I(1)	-C(1)	/	OM	=	58.5(2),	15.48(19),	15.8(2),	14.18(17),	4.5(2),	
	O(2)	-C(5)	/	OM	=	24.0(2),	30.2(2),	34.9(2),	2.4(2),	32.8(2),	
	O(3)	-C(6)	/	OM	=	49.8(2),	41.9(2),	62.0(2),	30.5(2),	50.2(2),	
	C(1)	-C(5)	/	OM	=	34.7(3),	18.1(2),	31.9(3),	61.4(2),	23.9(3),	
	-C(10)	/	OM	=	37.4(3),	18.0(2),	32.9(3),	2.1(2),	23.7(3),		
	C(4)	-C(7)	/	OM	=	1.9(3),	0.7(2),	1.3(2),	31.2(2),	0.7(2),	
	-C(8)	/	OM	=	2.0(3),	76.0(2),	44.7(3),	35.8(2),	65.0(3),		
	-C(10)	/	OM	=	59.0(3),	10.1(2),	20.0(3),	26.0(2),	0.6(3),		
	-C(11)	/	OM	=	54.1(3),	37.0(3),	59.5(3),	29.4(3),	45.8(3),		
	C(5)	-C(7)	/	OM	=	58.7(3),	11.0(2),	19.0(3),	7.2(2),	0.6(3),	
	-C(9)	/	OM	=	8.9(3),	76.1(2),	50.3(3),	49.6(2),	68.8(3),		
	C(6)	-C(7)	/	OM	=	1.9(3),	72.1(2),	40.8(2),	34.0(2),	61.1(3),	
	-C(8)	/	OM	=	2.0(3),	0.0(2),	1.3(3),	33.7(2),	0.7(3),		

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Ring Puckering Analysis

Ring Puckering Analysis (Cremer & Pople) - (e.s.d. following Norrestam, *Acta Cryst.* (1981), A37, 764-765)

Symmetrical Forms

References

5-Membered Rings : E : Envelope - Phi = k X 36
 T : Half Chair- Phi = k X 36 + 18
7-Membered Rings : C : Chair
 TC : Twist-Chair
 B : Boat
 TB : Twist-Boat

Definitions (All Values Rounded on Esd)

Dev - Deviation of Atom I from Cremer&Pople Plane (Defined Differently from Least-Squares Plane)
Cs(I),C2(I) - Mirror Plane and 2-Axis Asym. Par. for Atom I (See Duax et al., *Topics in Stereochemistry*,V-9, (1976) pp.271-383)
Cs(I-J),C2(I-J) - Asymmetry Parameters for Bond I-J
Tors(I-J) - Torsion Angle for Bond I-J
Descriptors for Torsion Angles

Torsion Angle Range		Full Descriptor	Short Descriptor	Angle Range of Subst.		Full Descriptor	Short Descriptor
0	TO 30 Deg	+ Syn-Periplanar	+sp	0	TO 30 Deg.	Axial	ax
30	to 90	+ Syn-Clinal	+sc	30	to 60	Bisectional	bi
90	to 150	+ Anti-Clinal	+ac	60	to 90	Equatorial	eq
150	to 180	+ Anti-Periplanar	+ap				
0	to -30	- Syn-Periplanar	-sp				
-30	to -90	- Syn-Clinal	-sc				
-90	to -150	- Anti-Clinal	-ac				
-150	to -180	- Anti-Periplanar	-ap				
*** NOTE *** - For Ring Puckering Comparisons: Make Sure that the Absolute Configuration, Pivot Atom and Cyclic Sense Agree.							
- The "RING AT1 AT2 AT3 ... ATn" Instruction Gives the User Explicit Choice of Pivot Atom (AT1) and Sense (AT2).							
- Use TRNS Instructions to Obtain the Required Absolute Configuration.							
- The Values of Theta and Phi [= Phi(2)] Depend on the Abs. Conf. and the Choice of the First and Second Ring Atom.							
- Alternatively, Appropriate Phase Shifts may be Applied to the Same Effect (see Below)							
For Correct Usage of C&P Puckering Parameters see also: D. Cremer, <i>Acta Cryst.</i> (1984). B40, 498-500.							
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4-Membered Ring (1) C(4) --> C(7) --> C(6) --> C(8) -->

Dev. (Ang) sp3 sp3 sp3
Cs(I)-Asym-Par (Deg) 0.026(4) -0.026(4) 0.026(4) -0.026(4)
C2(I)-Asym-Par (Deg) 0.1(3) 0.00 0.1(3) 0.00
Ring Bond Angle(Deg) 7.7(3) 7.7(3) 7.7(3) 7.7(3)
Tors(I-J) (Deg) 88.8(2) 89.4(3) 90.0(3) 91.5(3)
 -3.8(3) 3.8(3) -3.9(3) 3.9(3)

Cs(I-J)-Asym-Par (Deg) 7.7(4) 7.7(4) 7.7(4)
 C2(I-J)-Asym-Par (Deg) 0.1(4) 0.1(4) 0.1(4)
 Ring Bond Distance (Ang) 1.575(5) 1.530(5) 1.539(5)
 Weighted Average Ring Bond Distance = 1.5485(25, 97) Ang. - NOTE: 1st esd. Internal, 2nd esd External
 Weighted Average Abs. Torsion Angl. = 3.85(15, 3) Deg. see: e.g. Domenicano et al., Acta Cryst.(1975), B31, 221-234
 Angle between C(4) C(7) C(8) and C(7) C(6) C(8) = 174.6(4) deg.
 Angle between C(4) C(7) C(6) and C(4) C(6) C(8) = 174.5(4) deg.
 No C & P - Puckering Analysis since <Tau> = 3.8 < 5.0 Deg.
 Centroid Cg(1) : x , y , z 0.8319(2) 0.38398(15) 0.38802(8)
 XO, YO, ZO 7.0132(17) 4.5125(18) 8.5240(17)

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5-Membered Ring (2) C(1) --> C(5) --> C(7) --> C(4) --> C(10) -->

 Dev. (Ang) sp3 sp3 sp3
 Cs(I)-Asym-Par (Deg) -0.260(4) 0.215(4) -0.088(4) -0.072(4) 0.205(4)
 Cs(I)-Asym-Par (Deg) 1.4(3) 29.7(4) 48.6(4) 50.0(4) 32.4(4)
 C2(I)-Asym-Par (Deg) 67.3(4) 55.6(4) 22.7(4) 18.9(4) 53.2(4)
 Ring Bond Angle (Deg) 105.2(3) 101.3(3) 107.3(3) 105.1(3) 103.5(3)
 Tors (I-J) (Deg) 40.9(4) -25.4(3) 1.6(4) 23.5(3) -40.7(4)
 Cs(I-J)-Asym-Par (Deg) 50.0(4) 32.4(4) 1.4(3) 29.7(4) 48.6(4)
 C2(I-J)-Asym-Par (Deg) 18.9(4) 53.2(4) 67.3(4) 55.6(4) 22.7(4)
 Ring Bond Distance (Ang) 1.543(5) 1.537(5) 1.575(5) 1.537(5) 1.521(5)
 Weighted Average Ring Bond Distance = 1.5426(22, 89) Ang. - NOTE: 1st esd. Internal, 2nd esd External
 Weighted Average Abs. Torsion Angl. = 25.95(16, 631) Deg. see: e.g. Domenicano et al., Acta Cryst.(1975), B31, 221-234
 Cremer & Pople Puckering Parameters [D. Cremer & J.A. Pople, J.Amer.Chem.Soc., 97, (1975), 1354-1358]

 Q(2) = 0.4108(39) Ang., Phi(2) = 181.9(5) Deg
 *** NOTE *** - A Change of the Absolute Configuration Transforms Phi(2) into 180 + Phi(2).
 - A Cyclic Forward Shift of the Pivot Atom From At1 to At2 Transforms Phi(2) into Phi(2) + 144.
 - A Change of the Sense Transforms Phi(2) into 180 - Phi(2).
 Pseudorotation Parameters P and Tau(M), (Rao, Westhof & Sundaralingam, Acta Cryst (1981), A37, 421-425)

 P = 343.7(3) Degree, Tau(M) = 42.6(2) Deg. for Reference Bond C(1) --> C(5) [add 144 Deg. to P for each shift to next Bond]
 [Ring-Sequence Change of Sense : P ---->>> - P]

Note: DELTA [Defined in Altona,C.,Geise,H.J.,Romers,C.(1968). Tetrahedron, 24, 13-32] = 2 * P = 687.4 Deg.

Closest Pucker Descriptor: Envelope on C(1)

Conformational Analysis (G.G. Evans & J.A. Boeyens, Acta Cryst. (1989), B45, 581-590)

Coefficients of Primitive and Normalised Forms

M Primitive Coefficient Angular Value

CosForm 2 0.369 0.893 10.0

SinForm 0.044 0.107 11.0

Analysis of Ring Substituents

Bond Distance Angle with C&P Plane Normal

C(1) - I(1) 2.152(4) 74.17(19) Eq
 C(5) - O(2) 1.430(4) 60.0(2) Bi
 C(5) - C(9) 1.518(5) 13.6(2) Ax
 C(7) - C(6) 1.550(5) 17.5(2) Ax
 C(4) - C(8) 1.539(5) 13.7(2) Ax

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C(4) - C(11) 1.512(6) 53.3(3) Bi
 C(1) - H(1) 0.9792 1.28(15) Ax
 C(7) - H(7) 0.9795 51.93(15) Bi
 C(10) - H(10A) 0.9702 58.72(15) Bi
 C(10) - H(10B) 0.9701 13.15(15) Ax

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7-Membered Ring (3) C(1) --> C(5) --> C(7) --> C(6) --> C(8) --> C(4) --> C(10) -->

Dev. (Ang) sp3 sp3 sp3 sp3 sp3
 Cs(I)-Asym-Par (Deg) -0.675(4) 0.143(4) 0.619(4) -0.412(4) -0.455(4) 0.643(4) 0.137(4)
 C2(I)-Asym-Par (Deg) 2.6(2) 115.9(3) 48.9(3) 124.7(2) 120.8(2) 50.4(3) 115.9(3)
 Ring Bond Angle (Deg) 157.3(3) 57.5(3) 137.6(3) 90.1(3) 95.1(3) 136.4(3) 62.6(3)
 Tors (I-J) (Deg) 105.2(3) 101.3(3) 118.4(3) 90.0(3) 91.5(3) 113.7(3) 103.5(3)
 Cs(I-J)-Asym-Par (Deg) 40.9(4) 73.7(3) -105.6(3) -3.9(3) 109.8(3) -71.9(3) -40.7(4)
 C2(I-J)-Asym-Par (Deg) 120.8(2) 50.4(3) 115.9(3) 2.6(2) 115.9(3) 48.9(3) 124.7(2)
 Ring Bond Distance (Ang) 95.1(3) 136.4(3) 62.6(3) 157.3(3) 57.5(3) 137.6(3) 90.1(3)
 Weighted Average Ring Bond Distance = 1.5367(19, 35) Ang. - NOTE: 1st esd. Internal, 2nd esd External
 Weighted Average Abs. Torsion Angl. = 67.07(12, 999) Deg. see: e.g. Domenicano et al., Acta Cryst. (1975), B31, 221-234
 Cremer & Pople Puckering Parameters [D. Cremer & J.A. Pople, J.Amer.Chem.Soc., 97, (1975), 1354-1358]

Q(2) = 1.2908(37) Ang., Phi(2) = 179.59(17) Deg
 Q(3) = 0.0440(37) Ang., Phi(3) = 310(5) Deg
 Total Puckering Amplitude Q = 1.2916(37) Ang.
 Conformational Analysis (G.G. Evans & J.A. Boeyens, Acta Cryst. (1989), B45, 581-590)

Coefficients of Primitive and Normalised Forms

M Primitive Coefficient Angular Value

CosForm 2 1.250 0.936 14.0
 SinForm 0.042 0.031 13.0
 CosForm 3 0.039 0.029 24.0
 SinForm 0.005 0.004 25.0

:: Note: This is Very Similar to a B -Form

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Analysis of X-H...Cg(Pi-Ring) Interactions (H..Cg < 3.4 Ang. - Gamma < 30.0 Deg)

X--H(I) Res(I) Cg(J) [ARU(J)] H..Cg Transformed J-Plane P, Q, R, S H-Perp Gamma X-H..Cg X..Cg
C(1) -H(1) [1] -> Cg(1) [1555.01] 2.4710 0.5315 0.8335 0.1510 8.7755 2.458 6.00 81.60 2.522(4)

[1555] = X,Y,Z
Min or Max 2.471 2.458 6.00 81.60 2.522

The Cg(I) refer to the Ring Centre-of-Gravity numbers given in () in the Ring-Analysis above

Cg(I) x y z Xo Yo Zo
Cg(1) 0.8319(2) 0.38398(15) 0.38802(8) 7.0132(17) 4.5125(18) 8.5240(17)
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Table 0 - Crystal Data and Details of the Structure Determination
for: rosini

Crystal Data

Formula C9 H15 I O2
Formula Weight 282.11
Crystal System Orthorhombic
Space group (No. 61)
a, b, c [Angstrom] 8.4301(6) 11.7520(9) 21.9680(17)
V [Ang**3] 2176.4(3)
Z 8
D(calc) [g/cm**3] 1.722
Mu(MoKa) [/mm] 2.907
F(000) 1104
Crystal Size [mm] 0.30 x 0.40 x 0.80

Data Collection

Temperature (K) 293
Radiation [Angstrom] MoKa 0.71073
Theta Min-Max [Deg] 1.9, 30.0
Dataset -11: 11 ; -16: 16 ; -30: 30
Tot., Uniq. Data, R(int) 26880, 3165, 0.078
Observed data [I > 2.0 sigma(I)] 2479

Refinement

Nref, Npar 3165, 112
R, wR2, S 0.0586, 0.1749, 1.72
w = 1/[\s^2^(Fo^2^)+(0.0593P)^2^]
Max. and Av. Shift/Error where P=(Fo^2^+2Fc^2^)/3
Min. and Max. Resd. Dens. [e/Ang^3] 0.00, 0.00
-1.25, 1.64

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Notice

- PLATON Reference : Spek, A.L. (2003), J.Appl.Cryst. 36, 7-13
 - Output Values (Esd) may have been set to 99, 999 or 9999 to Avoid Format Overflow
 - Derived Parameter SU's (= Esd's) may be Incorrect in Cases where Covariances in the Atom Parameters should have been taken into Account (e.g. Those Involving Atoms That were Refined with Constraints)
 - ROUNDING, in particular of the Input Coordinate Data, may give deviating values for derived geometry parameters. However, differences should be within the associated esd-range.
 - PLATON is NOT a Finished Program. The Implementation of Additional Options is Planned. Some of the More Advanced Features are Experimental and may Contain Loose Ends.
 - The Communication of Glitches Encountered will be Appreciated: E-mail: a.l.spek@chem.uu.nl
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Summary and Remarks : N = NOTE, W = WARNING, E = ERROR

W: NOMOVE option used.
 :: >>> WARNING: 'CONNECTED INPUT SET' is assumed 'TRUE'
 :: >>> Network Analysis may be INCORRECT when FALSE

:: NORMAL END of PLATON : 26 Pages on:
 :: test.lis (ASCII, 132 Characters Wide)
 :: test.lps (PostScript Version of .lis)