Table 2. Anisotropic Displacement Parameters (continued)

atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(14)	0.020(5)	0.021(6)	0.023(7)	0.005(5)	0.013(6)	0.001(6)
C(15)	0.033(6)	0.024(6)	0.023(6)	0.003(5)	-0.001(5)	0.004(6)
C(16)	0.041(7)	0.036(7)	0.019(6)	-0.020(6)	-0.001(5)	-0.001(6)
C(17)	0.041(7)	0.043(7)	0.026(7)	-0.015(6)	0.000(5)	-0.011(7)
C(18)	0.041(7)	0.042(7)	0.009(6)	-0.003(6)	0.011(5)	-0.009(5)
C(19)	0.050(7)	0.033(7)	0.026(6)	-0.006(6)	0.020(6)	-0.004(6)
C(20)	0.044(7)	0.024(7)	0.033(7)	-0.008(6)	0.015(6)	-0.001(6)
C(21)	0.020(7)	0.027(6)	0.036(8)	0.009(5)	0.009(6)	0.005(6)
C(22)	0.018(7)	0.021(6)	0.063(8)	0.001(5)	0.019(6)	-0.005(6)
C(23)	0.020(7)	0.036(7)	0.080(10)	0.000(6)	0.025(7)	0.007(8)
C(24)	0.033(9)	0.069(10)	0.056(10)	-0.004(7)	0.015(7)	0.031(8)
C(25)	0.031(7)	0.079(10)	0.055(8)	0.010(7)	0.002(7)	0.016(8)
C(26)	0.018(6)	0.070(9)	0.044(8)	-0.006(6)	0.002(6)	0.013(8)
C(27)	0.037(9)	0.29(3)	0.10(1)	0.014(13)	-0.022(10)	0.09(2)
C(28)	0.0243	0.0196	0.0432	0.0132	-0.0079	-0.0120

The general temperature factor expression:

$\exp(-2\pi^2(a^{*2}U_{11}h^2 + b^{*2}U_{22}k^2 + c^{*2}U_{33}l^2 + 2a^*b^*U_{12}hk + 2a^*c^*U_{13}hl + 2b^*c^*U_{23}kl))$

Table 3. Bond Lengths(Å)

atom	atom	distance	atom	atom	distance
Br(1)	C(18)	1.888(10)	O(1)	C(1)	1.417(12)
O(1)	C(14)	1.358(12)	O(2)	C(14)	1.209(12)
O(3)	C(24)	1.37(2)	O(3)	C(27)	1.42(2)
O(4)	C(11)	1.230(13)	O(5)	C(13)	1.348(12)
O(5)	C(28)	1.437(12)	N(1)	C(14)	1.364(13)
N(1)	C(15)	1.423(13)	N(1)	H(1)	0.95
C(1)	C(2)	1.51(1)	C(1)	C(13)	1.52(1)
C(1)	H(17)	0.95	C(2)	C(3)	1.57(1)
C(2)	C(10)	1.54(2)	C(2)	H(3)	0.95
C(3)	C(4)	1.57(1)	C(3)	C(9)	1.50(1)
C(3)	C(21)	1.57(2)	C(4)	C(5)	1.52(2)
C(4)	H(18)	0.95	C(4)	H(19)	0.95
C(5)	C(6)	1.50(2)	C(5)	H(20)	0.95
C(5)	H(21)	0.95	C(6)	C(7)	1.56(2)
C(6)	H(5)	0.95	C(6)	H(6)	0.95
C(7)	C(8)	1.50(2)	C(7)	H(22)	0.95
C(7)	H(23)	0.95	C(8)	C(9)	1.54(2)
C(8)	H(24)	0.95	C(8)	H(25)	0.95
C(9)	C(10)	1.56(1)	C(9)	H(4)	0.95
C(10)	C(11)	1.50(2)	C(10)	H(26)	0.95

Table 3. Bond Lengths(Å) (continued)

atom	atom	distance	atom	atom	distance
C(11)	C(12)	1.48(2)	C(12)	C(13)	1.34(1)
C(12)	H(30)	0.95	C(15)	C(16)	1.37(2)
C(15)	C(20)	1.38(2)	C(16)	C(17)	1.37(2)
C(16)	H(2)	0.95	C(17)	C(18)	1.36(2)
C(17)	H(27)	0.95	C(18)	C(19)	1.41(2)
C(19)	C(20)	1.37(2)	C(19)	H(28)	0.95
C(20)	H(29)	0.95	C(21)	C(22)	1.42(2)
C(21)	C(26)	1.35(2)	C(22)	C(23)	1.36(2)
C(22)	H(10)	0.95	C(23)	C(24)	1.32(2)
C(23)	H(9)	0.95	C(24)	C(25)	1.40(2)
C(25)	C(26)	1.45(2)	C(25)	H(8)	0.95
C(26)	H(7)	0.95	C(27)	H(14)	0.95
C(27)	H(15)	0.95	C(27)	H(16)	0.95
C(28)	H(11)	0.95	C(28)	H(12)	0.95
C(28)	H(13)	0.95			

Table 4. Bond Angles(°)

atom	atom	atom	angle	atom	atom	atom	angle
C(1)	O(1)	C(14)	114.6(7)	C(24)	O(3)	C(27)	119.6(11)
C(13)	O(5)	C(28)	117.5(8)	C(14)	N(1)	C(15)	126.8(9)
C(14)	N(1)	H(1)	116.6	C(15)	N(1)	H(1)	116.6
O(1)	C(1)	C(2)	111.4(8)	O(1)	C(1)	C(13)	109.8(8)
O(1)	C(1)	H(17)	107.7	C(2)	C(1)	C(13)	112.2(8)
C(2)	C(1)	H(17)	107.7	C(13)	C(1)	H(17)	107.7
C(1)	C(2)	C(3)	125.3(8)	C(1)	C(2)	C(10)	116.2(8)
C(1)	C(2)	H(3)	108.0	C(3)	C(2)	C(10)	89.4(8)
C(3)	C(2)	H(3)	108.0	C(10)	C(2)	H(3)	108.0
C(2)	C(3)	C(4)	116.6(8)	C(2)	C(3)	C(9)	90.2(8)
C(2)	C(3)	C(21)	109.8(8)	C(4)	C(3)	C(9)	115.5(9)
C(4)	C(3)	C(21)	107.0(8)	C(9)	C(3)	C(21)	117.4(9)
C(3)	C(4)	C(5)	114.2(9)	C(3)	C(4)	H(18)	108.3
C(3)	C(4)	H(19)	108.3	C(5)	C(4)	H(18)	108.3
C(5)	C(4)	H(19)	108.3	H(18)	C(4)	H(19)	109.5
C(4)	C(5)	C(6)	117.0(10)	C(4)	C(5)	H(20)	107.6
C(4)	C(5)	H(21)	107.6	C(6)	C(5)	H(20)	107.6
C(6)	C(5)	H(21)	107.6	H(20)	C(5)	H(21)	109.5
C(5)	C(6)	C(7)	116.8(10)	C(5)	C(6)	H(5)	107.6
C(5)	C(6)	H(6)	107.6	C(7)	C(6)	H(5)	107.6

Table 4. Bond Angles(°) (continued)

atom	atom	atom	angle	atom	atom	atom	angle
C(7)	C(6)	H(6)	107.6	H(5)	C(6)	H(6)	109.5
C(6)	C(7)	C(8)	112.6(10)	C(6)	C(7)	H(22)	108.7
C(6)	C(7)	H(23)	108.7	C(8)	C(7)	H(22)	108.7
C(8)	C(7)	H(23)	108.7	H(22)	C(7)	H(23)	109.5
C(7)	C(8)	C(9)	115.2(10)	C(7)	C(8)	H(24)	108.0
C(7)	C(8)	H(25)	108.0	C(9)	C(8)	H(24)	108.0
C(9)	C(8)	H(25)	108.0	H(24)	C(8)	H(25)	109.5
C(3)	C(9)	C(8)	120.6(9)	C(3)	C(9)	C(10)	90.9(8)
C(3)	C(9)	H(4)	109.0	C(8)	C(9)	C(10)	116.9(9)
C(8)	C(9)	H(4)	109.0	C(10)	C(9)	H(4)	109.0
C(2)	C(10)	C(9)	89.0(7)	C(2)	C(10)	C(11)	117.3(9)
C(2)	C(10)	H(26)	111.5	C(9)	C(10)	C(11)	114.3(9)
C(9)	C(10)	H(26)	111.5	C(11)	C(10)	H(26)	111.5
O(4)	C(11)	C(10)	121.6(9)	O(4)	C(11)	C(12)	119.4(9)
C(10)	C(11)	C(12)	118.9(9)	C(11)	C(12)	C(13)	120.4(10)
C(11)	C(12)	H(30)	119.8	C(13)	C(12)	H(30)	119.8
O(5)	C(13)	C(1)	111.6(8)	O(5)	C(13)	C(12)	124.8(9)
C(1)	C(13)	C(12)	123.3(9)	O(1)	C(14)	O(2)	124.6(9)
O(1)	C(14)	N(1)	109.4(8)	O(2)	C(14)	N(1)	126.0(9)
N(1)	C(15)	C(16)	117.1(9)	N(1)	C(15)	C(20)	124.0(9)

Table 4. Bond Angles(°) (continued)

atom	atom	atom	angle	atom	atom	atom	angle
C(16)	C(15)	C(20)	118.8(10)	C(15)	C(16)	C(17)	120.6(10)
C(15)	C(16)	H(2)	119.7	C(17)	C(16)	H(2)	119.7
C(16)	C(17)	C(18)	120.4(11)	C(16)	C(17)	H(27)	119.8
C(18)	C(17)	H(27)	119.8	Br(1)	C(18)	C(17)	120.6(8)
Br(1)	C(18)	C(19)	118.9(8)	C(17)	C(18)	C(19)	120.2(10)
C(18)	C(19)	C(20)	118.1(10)	C(18)	C(19)	H(28)	121.0
C(20)	C(19)	H(28)	121.0	C(15)	C(20)	C(19)	121.8(10)
C(15)	C(20)	H(29)	119.1	C(19)	C(20)	H(29)	119.1
C(3)	C(21)	C(22)	122.4(10)	C(3)	C(21)	C(26)	118.3(9)
C(22)	C(21)	C(26)	119.2(10)	C(21)	C(22)	C(23)	118.2(11)
C(21)	C(22)	H(10)	120.9	C(23)	C(22)	H(10)	120.9
C(22)	C(23)	C(24)	124.7(12)	C(22)	C(23)	H(9)	117.7
C(24)	C(23)	H(9)	117.7	O(3)	C(24)	C(23)	119.0(12)
O(3)	C(24)	C(25)	121.7(13)	C(23)	C(24)	C(25)	119.3(12)
C(24)	C(25)	C(26)	117.5(12)	C(24)	C(25)	H(8)	121.2
C(26)	C(25)	H(8)	121.2	C(21)	C(26)	C(25)	121.0(11)
C(21)	C(26)	H(7)	119.5	C(25)	C(26)	H(7)	119.5
O(3)	C(27)	H(14)	109.5	O(3)	C(27)	H(15)	109.5
O(3)	C(27)	H(16)	109.5	H(14)	C(27)	H(15)	109.5
H(14)	C(27)	H(16)	109.5	H(15)	C(27)	H(16)	109.5

Table 4. Bond Angles(°) (continued)

atom	atom	atom	angle	atom	atom	atom	angle
O(5)	C(28)	H(11)	109.5	O(5)	C(28)	H(12)	109.5
O(5)	C(28)	H(13)	109.5	H(11)	C(28)	H(12)	109.5
H(11)	C(28)	H(13)	109.5	H(12)	C(28)	H(13)	109.5

Table 5. Torsion Angles(°)

atom	atom	atom	atom	angle	atom	atom	atom	atom	angle
Br(1)	C(18)	C(17)	C(16)	177.6(9)	Br(1)	C(18)	C(19)	C(20)	-178.9(9)
O(1)	C(1)	C(2)	C(3)	51.3(12)	O(1)	C(1)	C(2)	C(10)	160.9(8)
O(1)	C(1)	C(13)	O(5)	28.3(11)	O(1)	C(1)	C(13)	C(12)	-158.1(9)
O(1)	C(14)	N(1)	C(15)	-179.1(9)	O(2)	C(14)	O(1)	C(1)	-6.5(13)
O(2)	C(14)	N(1)	C(15)	3.7(16)	O(3)	C(24)	C(23)	C(22)	-178.0(12)
O(3)	C(24)	C(25)	C(26)	179.4(13)	O(4)	C(11)	C(10)	C(2)	173.8(9)
O(4)	C(11)	C(10)	C(9)	-84.0(12)	O(4)	C(11)	C(12)	C(13)	-167.5(10)
O(5)	C(13)	C(1)	C(2)	152.8(8)	O(5)	C(13)	C(12)	C(11)	180.0(9)
N(1)	C(14)	O(1)	C(1)	176.2(8)	N(1)	C(15)	C(16)	C(17)	-177.9(10)
N(1)	C(15)	C(20)	C(19)	176.5(10)	C(1)	C(2)	C(3)	C(4)	9.2(14)
C(1)	C(2)	C(3)	C(9)	128.0(10)	C(1)	C(2)	C(3)	C(21)	-112.6(10)
C(1)	C(2)	C(10)	C(9)	-135.3(9)	C(1)	C(2)	C(10)	C(11)	-18.2(13)
C(1)	C(13)	O(5)	C(28)	170.0(8)	C(1)	C(13)	C(12)	C(11)	7.3(15)
C(2)	C(1)	O(1)	C(14)	141.2(8)	C(2)	C(1)	C(13)	C(12)	-33.6(13)
C(2)	C(3)	C(4)	C(5)	173.9(9)	C(2)	C(3)	C(9)	C(8)	-128.0(10)
C(2)	C(3)	C(9)	C(10)	-5.6(7)	C(2)	C(3)	C(21)	C(22)	-127.6(10)
C(2)	C(3)	C(21)	C(26)	52.8(13)	C(2)	C(10)	C(9)	C(3)	5.7(8)
C(2)	C(10)	C(9)	C(8)	131.1(9)	C(2)	C(10)	C(11)	C(12)	-8.5(14)
C(3)	C(2)	C(1)	C(13)	-72.3(12)	C(3)	C(2)	C(10)	C(9)	-5.5(7)
C(3)	C(2)	C(10)	C(11)	111.5(9)	C(3)	C(4)	C(5)	C(6)	-80.1(12)

Table 5. Torsion Angles(°) (continued)

atom	atom	atom	atom	angle	atom	atom	atom	atom	angle
C(3)	C(9)	C(8)	C(7)	-62.7(13)	C(3)	C(9)	C(10)	C(11)	-113.9(9)
C(3)	C(21)	C(22)	C(23)	179.1(10)	C(3)	C(21)	C(26)	C(25)	-177.6(11)
C(4)	C(3)	C(2)	C(10)	-113.1(9)	C(4)	C(3)	C(9)	C(8)	-8.2(14)
C(4)	C(3)	C(9)	C(10)	114.1(9)	C(4)	C(3)	C(21)	C(22)	105.0(11)
C(4)	C(3)	C(21)	C(26)	-74.5(13)	C(4)	C(5)	C(6)	C(7)	58.7(14)
C(5)	C(4)	C(3)	C(9)	69.9(12)	C(5)	C(4)	C(3)	C(21)	-62.8(11)
C(5)	C(6)	C(7)	C(8)	-61.5(15)	C(6)	C(7)	C(8)	C(9)	83.3(13)
C(7)	C(8)	C(9)	C(10)	-171.4(9)	C(8)	C(9)	C(3)	C(21)	119.5(11)
C(8)	C(9)	C(10)	C(11)	11.5(13)	C(9)	C(3)	C(2)	C(10)	5.7(8)
C(9)	C(3)	C(21)	C(22)	-26.6(14)	C(9)	C(3)	C(21)	C(26)	153.8(11)
C(9)	C(10)	C(11)	C(12)	93.7(11)	C(10)	C(2)	C(1)	C(13)	37.3(11)
C(10)	C(2)	C(3)	C(21)	125.1(8)	C(10)	C(9)	C(3)	C(21)	-118.2(9)
C(10)	C(11)	C(12)	C(13)	14.8(15)	C(12)	C(13)	O(5)	C(28)	-3.4(14)
C(13)	C(1)	O(1)	C(14)	-93.8(9)	C(14)	N(1)	C(15)	C(16)	175.3(10)
C(14)	N(1)	C(15)	C(20)	-3.8(17)	C(15)	C(16)	C(17)	C(18)	-1.3(17)
C(15)	C(20)	C(19)	C(18)	3.8(17)	C(16)	C(15)	C(20)	C(19)	-2.6(17)
C(16)	C(17)	C(18)	C(19)	2.6(17)	C(17)	C(16)	C(15)	C(20)	1.3(16)
C(17)	C(18)	C(19)	C(20)	-3.8(17)	C(21)	C(22)	C(23)	C(24)	-0.5(18)
C(21)	C(26)	C(25)	C(24)	-2.5(21)	C(22)	C(21)	C(26)	C(25)	2.8(19)
C(22)	C(23)	C(24)	C(25)	0.8(21)	C(23)	C(22)	C(21)	C(26)	-1.4(16)

Table 5. Torsion Angles(°) (continued)

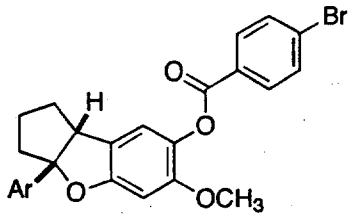
atom	atom	atom	atom	angle	atom	atom	atom	atom	angle
C(23)	C(24)	O(3)	C(27)	-177.3(17)	C(23)	C(24)	C(25)	C(26)	0.6(21)
C(25)	C(24)	O(3)	C(27)	3.9(23)					

Ref. No.: Crystal-168

X-RAY STRUCTURE REPORT

for

Prof. T. Engler



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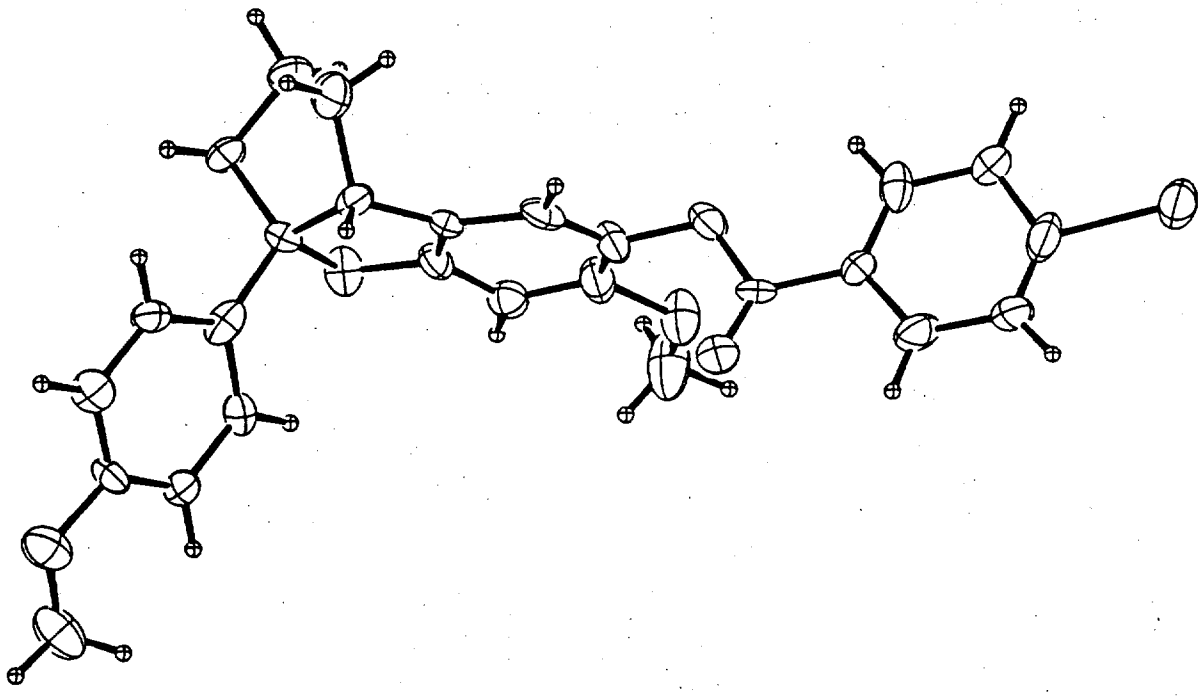
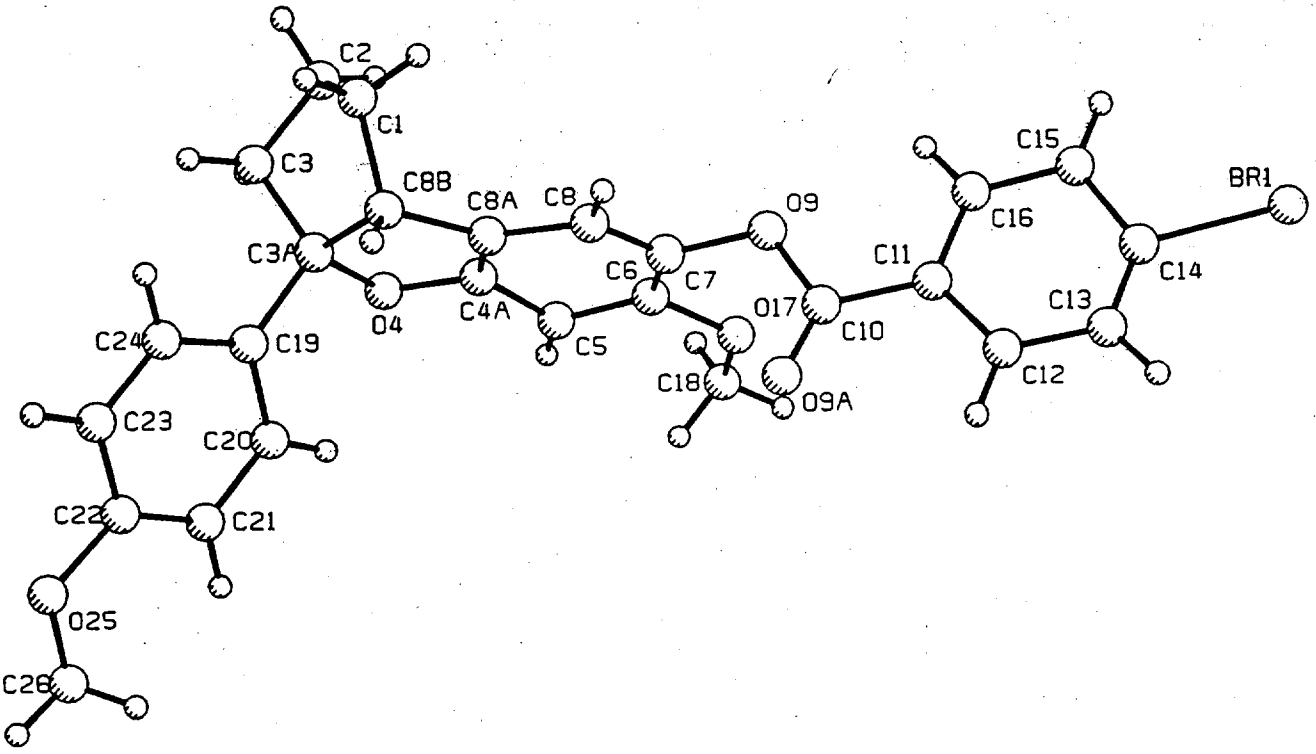
8-NOV-94



crustal-168



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INTRODUCTION

The absolute configuration of the molecule was determined using the anomalous dispersion of the Br-atom. The results are shown below:

	Reported Structure	Enantiomorph
No. Reflections	815	815
No. Parameters	288	288
R-factor	0.061	0.063
wR-factor	0.080	0.082
Goodness fit	1.45	1.48

According to the Hamilton R-factor test (Hamilton, W. C., *Acta Cryst.*, **18**, 502 (1965)), the model stands at more than 99.5% confidence level.

EXPERIMENTAL

DATA COLLECTION

A colorless prism crystal of $BrC_{26}H_{23}O_5$ having approximate dimensions of 0.300 X 0.300 X 0.200 mm was mounted on a glass fiber. All measurements were made on a Rigaku AFC5R diffractometer with graphite monochromated Cu Kα radiation and a 12KW rotating anode generator.

Cell constants and an orientation matrix for data collection, obtained from a least-squares refinement using the setting angles of 25 carefully centered reflections in the range $35.00 < 2\theta < 50.00°$ corresponded to a monoclinic cell with dimensions:

$a = 19.492\ (3)Å$
$b = 10.053\ (2)Å$ $\quad \beta = 98.92\ (3)°$
$c = 12.145\ (2)Å$
$V = 2351\ (1)Å^3$

For $Z = 4$ and F.W. = 495.37, the calculated density is 1.399 g/cm^3. Based on the systematic absences of:

hkl: $h+k \neq 2n$

packing considerations, a statistical analysis of intensity distribution, and the successful solution and refinement of the structure, the space group was determined to be:

C2 (#5)

The data were collected at a temperature of 23 ± 1°C using the ω-2θ scan technique to a maximum 2θ value of 112.7°. Omega scans of several intense reflections, made prior to data collection, had an average width at half-height of 0.80° with a take-off angle of 6.0°. Scans of (1.78 + 0.30 tan θ)° were made at a speed of 16.0°/min (in omega). The weak reflections ($I < 10.0\sigma(I)$) were rescanned (maximum of 2 rescans) and the counts were accumulated to assure good counting statistics. Stationary background counts were recorded on each side of the reflection. The ratio of peak counting time to background counting time was 2:1. The diameter of the incident beam collimator was 0.5 mm and the crystal to detector distance was 285.0 mm.

DATA REDUCTION

Of the 1717 reflections which were collected, 1658 were unique (R_{int} = .111). The intensities of three representative reflections which were measured after every 150 reflections remained constant throughout data collection indicating crystal and electronic stability (no decay correction was applied).

The linear absorption coefficient for Cu Kα is 26.6 cm^{-1}. An empirical absorption correction, using the program DIFABS[3], was applied which resulted in transmission factors ranging from 0.72 to 1.18. The data were corrected for Lorentz and polarization effects.

STRUCTURE SOLUTION AND REFINEMENT

The structure was solved by direct methods[4]. The non-hydrogen atoms were refined anisotropically. The final cycle of full-matrix least-squares refinement[5] was based on 815 observed reflections ($I > 3.00\sigma(I)$) and 288 variable parameters and converged (largest parameter shift was 0.08 times its esd) with unweighted and weighted agreement factors of:

$$R = \Sigma \, ||Fo| - |Fc|| \, / \, \Sigma \, |Fo| = 0.061$$

$$R_w = [(\Sigma \, w \, (|Fo| - |Fc|)^2 \, / \, \Sigma \, w \, Fo^2)]^{1/2} = 0.080$$

The standard deviation of an observation of unit weight[6] was 1.45. The weighting scheme was based on counting statistics and included a factor (p = 0.10) to downweight the intense reflections[2]. Plots of $\Sigma \, w \, (|Fo| - |Fc|)^2$ versus |Fo|, reflection order in data collection, $\sin\theta/\lambda$, and various classes of indices showed no unusual trends. The maximum and minimum peaks on the final difference Fourier map corresponded to 0.48 and -0.30 $e^-/Å^3$, respectively.

Neutral atom scattering factors were taken from Cromer and Waber[7]. Anomalous dispersion effects were included in Fcalc[8]; the values for Δf′ and Δf″ were those of Cromer[9]. All calculations were performed using the TEXSAN[10] crystallographic software package of Molecular Structure Corporation.

References

(1) PLUTO:
Motherwell,S. & Clegg,W.; PLUTO. Program for plotting molecular and crystal structures. Univ. of Cambridge, England (1978).

(2) ORTEP:
Johnson,C.K.; ORTEPII. Report ORNL-5138. Oak Ridge National Laboratory, Oak Ridge, Tennessee (1976).

(3) DIFABS:
Walker & Stuart, Acta Cryst. A39, 158-166, (1983).

(4) Structure Solution Methods:
PHASE
Calbrese,J.C.; PHASE - Patterson Heavy Atom Solution Extractor. Univ. of Wisconsin-Madison, Ph.D. Thesis (1972).
DIRDIF
Beurskens,P.T.; DIRDIF: Direct Methods for Difference Structures - an automatic procedure for phase extension and refinement of difference structure factors. Technical Report 1984/1 Crystallography Laboratory, Toernooiveld, 6525 Ed Nijmegen, Netherlands.

(5) Least-Squares:
Function minimized: $\Sigma\, w\,(|Fo| - |Fc|)^2$
where: $w = 4Fo^2/\sigma^2(Fo^2)$
$\sigma^2(Fo^2) = [S^2(C+R^2B) + (pFo^2)^2]/Lp^2$
S = Scan rate
C = Total Integrated Peak Count
R = Ratio of Scan Time to background counting time.
B = Total Background Count
Lp = Lorentz-polarization factor
p = p-factor

(6) Standard deviation of an observation of unit weight:

$$[\Sigma\, w(|Fo| - |Fc|)^2/(No - Nv)]^{1/2}$$

where: No = number of observations
Nv = number of variables

(7) Cromer,D.T. & Waber,J.T.; "International Tables for X-ray Crystallography", Vol. IV, The Kynoch Press, Birmingham, England, Table 2.2 A (1974).

(8) Ibers,J.A. & Hamilton,W.C.; Acta Crystallogr., 17, 781 (1964).

(9) D.T. Cromer, "International Tables for X-ray

Crystallography", Vol/ IV, The Kynoch Press, Birmingham, England, Table 2.3.1 (1974).

(10) TEXSAN - TEXRAY Structure Analysis Package, Molecular Structure Corporation (1985).

EXPERIMENTAL DETAILS

A. Crystal Data

Empirical Formula	$BrC_{26}H_{23}O_5$
Formula Weight	495.37
Crystal Color, Habit	colorless, prism
Crystal Dimensions (mm)	0.300 X 0.300 X 0.200
Crystal System	monoclinic
No. Reflections Used for Unit Cell Determination (2θ range)	25 (35.0 - 50.0°)
Omega Scan Peak Width at Half-height	0.80
Lattice Parameters:	a = 19.492 (3)Å b = 10.053 (2)Å c = 12.145 (2)Å β = 98.92 (3)° V = 2351 (1)Å^3
Space Group	C2 (#5)
Z value	4
D_{calc}	1.399 g/cm^3
F_{000}	1016
$\mu_{(CuK\alpha)}$	26.62 cm^{-1}

B. Intensity Measurements

Diffractometer	Rigaku AFC5R
Radiation	CuKα (λ = 1.54178 Å)
Temperature	23°C
Attenuators	Zr foil (factors: 4.1, 14.5, 54.4)
Take-off Angle	6.0°

Detector Aperture	6.0 mm horizontal 6.0 mm vertical
Crystal to Detector Distance	40 cm
Scan Type	ω-2θ
Scan Rate	16.0°/min (in omega) (2 rescans)
Scan Width	(1.78 + 0.30 tanθ)°
$2\theta_{max}$	112.7°
No. of Reflections Measured	Total: 1717 Unique: 1658 (R_{int} = .111)
Corrections	Lorentz-polarization Absorption (trans. factors: 0.72 - 1.18)

C. Structure Solution and Refinement

Structure Solution	Patterson Method
Refinement	Full-matrix least-squares
Function Minimized	Σ w (|Fo| - |Fc|)^2
Least-squares Weights	$4Fo^2/\sigma^2(Fo^2)$
p-factor	0.10
Anomalous Dispersion	All non-hydrogen atoms
No. Observations (I>3.00σ(I))	815
No. Variables	288
Reflection/Parameter Ratio	2.83
Residuals: R; R_w	0.061; 0.080
Goodness of Fit Indicator	1.45
Max Shift/Error in Final Cycle	0.08
Maximum Peak in Final Diff. Map	0.48 $e^-/Å^3$
Minimum Peak in Final Diff. Map	-0.30 $e^-/Å^3$

Positional parameters for crystal-168

atom	x	y	z
Br(1)	0.4147(2)	0.9643	0.9141(2)
C(1)	0.222(1)	0.792(4)	-0.069(2)
C(2)	0.155(2)	0.725(3)	-0.101(2)
C(3)	0.111(1)	0.821(2)	-0.169(2)
C(3A)	0.129(1)	0.948(3)	-0.104(1)
O(4)	0.0819(5)	0.965(2)	-0.0200(9)
C(4A)	0.123(1)	0.961(3)	0.082(1)
C(5)	0.092(1)	0.996(2)	0.178(2)
C(6)	0.140(1)	0.982(3)	0.277(2)
C(7)	0.207(1)	0.963(3)	0.278(1)
C(8)	0.233(1)	0.946(3)	0.186(2)
C(8A)	0.190(1)	0.946(2)	0.080(1)
C(8B)	0.205(1)	0.930(2)	-0.037(1)
O(9)	0.2506(6)	0.948(2)	0.382(1)
O(9A)	0.283(1)	1.163(2)	0.384(1)
C(10)	0.281(1)	1.062(3)	0.431(2)
C(11)	0.314(1)	1.039(3)	0.547(2)
C(12)	0.343(1)	1.141(3)	0.613(2)
C(13)	0.375(2)	1.121(3)	0.720(3)
C(14)	0.371(1)	1.005(4)	0.763(2)
C(15)	0.346(2)	0.889(3)	0.705(2)
C(16)	0.313(1)	0.914(3)	0.596(2)
O(17)	0.1207(8)	1.016(2)	0.381(1)
C(18)	0.056(2)	1.047(4)	0.382(2)

Positional parameters for crystal-168

atom	x	y	z
C(19)	0.122(1)	1.076(3)	-0.175(2)
C(20)	0.080(1)	1.182(3)	-0.147(1)
C(21)	0.070(1)	1.294(2)	-0.216(2)
C(22)	0.098(1)	1.303(2)	-0.310(2)
C(23)	0.137(1)	1.204(3)	-0.339(2)
C(24)	0.148(1)	1.094(2)	-0.271(2)
O(25)	0.091(1)	1.411(2)	-0.385(2)
C(26)	0.051(1)	1.508(3)	-0.360(2)
H(1)	0.2482	0.7456	-0.0065
H(2)	0.2480	0.7913	-0.1285
H(3)	0.1605	0.6474	-0.1440
H(4)	0.1353	0.7011	-0.0374
H(5)	0.1223	0.8295	-0.2424
H(6)	0.0630	0.8027	-0.1735
H(7)	0.0444	1.0173	0.1766
H(8)	0.2821	0.9325	0.1897
H(9)	0.2358	0.9940	-0.0556
H(10)	0.3399	1.2294	0.5817
H(11)	0.3991	1.1924	0.7637
H(12)	0.3488	0.8025	0.7371
H(13)	0.2900	0.8430	0.5526
H(14)	0.0505	1.0748	0.4555
H(15)	0.0440	1.1221	0.3327
H(16)	0.0272	0.9757	0.3590
H(17)	0.0588	1.1810	-0.0821

Positional parameters for crystal-168

atom	x	y	z
H(18)	0.0426	1.3662	-0.1962
H(19)	0.1563	1.2088	-0.4064
H(20)	0.1766	1.0239	-0.2921
H(21)	0.0687	1.5436	-0.2888
H(22)	0.0511	1.5788	-0.4144
H(23)	0.0050	1.4789	-0.3616

U values for crystal-168

ATOM	U11	U22	U33	U12	U13	U23
Br(1)	0.168(3)	0.164(3)	0.082(1)	0.069(3)	-0.023(1)	-0.036(2)
C(1)	0.12(2)	0.13(3)	0.07(1)	0.00(2)	0.04(1)	-0.01(2)
C(2)	0.22(3)	0.03(2)	0.10(2)	0.01(2)	0.02(2)	0.00(1)
C(3)	0.11(2)	0.04(2)	0.10(2)	-0.02(1)	-0.00(2)	-0.02(2)
C(3A)	0.08(1)	0.05(1)	0.06(1)	0.02(1)	-0.01(1)	0.01(1)
O(4)	0.049(6)	0.11(1)	0.072(7)	-0.02(1)	0.019(6)	0.02(1)
C(4A)	0.07(1)	0.07(1)	0.05(1)	0.02(2)	-0.01(1)	-0.00(1)
C(5)	0.08(1)	0.07(2)	0.10(2)	0.00(1)	-0.01(1)	0.01(1)
C(6)	0.08(2)	0.10(2)	0.08(1)	0.02(2)	-0.00(1)	0.02(2)
C(7)	0.07(2)	0.10(2)	0.05(1)	0.02(2)	0.01(1)	0.02(2)
C(8)	0.05(1)	0.07(2)	0.12(2)	0.02(1)	-0.01(1)	0.01(2)
C(8A)	0.05(1)	0.05(1)	0.06(1)	0.03(1)	-0.005(8)	-0.00(1)
C(8B)	0.09(1)	0.05(2)	0.06(1)	0.00(1)	0.02(1)	-0.01(1)
O(9)	0.09(1)	0.06(1)	0.077(8)	0.02(1)	-0.009(7)	0.01(1)
O(9A)	0.15(1)	0.07(1)	0.10(1)	0.00(1)	-0.01(1)	-0.00(1)
C(10)	0.11(2)	0.03(2)	0.08(2)	0.03(1)	0.01(1)	-0.01(1)
C(11)	0.08(2)	0.05(2)	0.06(2)	-0.02(1)	-0.01(1)	-0.01(1)

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U values for crystal-168

ATOM	U11	U22	U33	U12	U13	U23
C(12)	0.12(2)	0.06(2)	0.12(2)	0.02(2)	-0.02(2)	-0.02(2)
C(13)	0.17(3)	0.08(2)	0.10(2)	0.03(2)	-0.05(2)	-0.03(2)
C(14)	0.12(2)	0.14(3)	0.05(1)	0.03(2)	-0.00(1)	-0.03(2)
C(15)	0.16(3)	0.09(2)	0.06(2)	0.03(2)	0.00(2)	-0.01(2)
C(16)	0.08(2)	0.13(3)	0.05(1)	0.01(2)	0.01(1)	-0.01(2)
O(17)	0.08(1)	0.17(2)	0.068(9)	0.03(1)	0.028(8)	-0.00(1)
C(18)	0.13(2)	0.26(5)	0.07(1)	0.02(3)	0.03(2)	-0.01(2)
C(19)	0.04(1)	0.11(2)	0.06(2)	0.00(1)	0.02(1)	-0.03(2)
C(20)	0.08(2)	0.10(2)	0.05(1)	-0.01(2)	0.01(1)	0.02(1)
C(21)	0.06(1)	0.07(2)	0.08(2)	0.02(1)	0.01(1)	-0.01(1)
C(22)	0.08(1)	0.07(2)	0.05(1)	0.02(1)	0.01(1)	0.02(1)
C(23)	0.10(2)	0.07(2)	0.09(1)	-0.02(2)	0.05(1)	0.01(2)
C(24)	0.11(2)	0.05(2)	0.08(1)	-0.00(1)	0.02(1)	-0.01(1)
O(25)	0.09(1)	0.09(2)	0.13(1)	-0.01(1)	0.01(1)	0.02(1)
C(26)	0.09(2)	0.13(3)	0.12(2)	0.01(2)	0.03(2)	0.06(2)
H(1)	0.1245					
H(2)	0.1245					
H(3)	0.1414					

U values for crystal-168

ATOM	U11	U22	U33	U12	U13	U23
H(4)	0.1414					
H(5)	0.1009					
H(6)	0.1009					
H(7)	0.0903					
H(8)	0.0935					
H(9)	0.0808					
H(10)	0.1242					
H(11)	0.1504					
H(12)	0.1180					
H(13)	0.1081					
H(14)	0.1834					
H(15)	0.1834					
H(16)	0.1834					
H(17)	0.0916					
H(18)	0.0852					
H(19)	0.1007					
H(20)	0.0918					
H(21)	0.1438					

U values for crystal-168

ATOM	U11	U22	U33	U12	U13	U23
H(22)	0.1438					
H(23)	0.1438					

Intramolecular Distances

atom	atom	distance	atom	atom	distance
BR1	C14	1.94(2)	C8B	H9	0.932
C1	C2	1.47(4)	O9	C10	1.38(3)
C1	C8B	1.49(4)	O9A	C10	1.17(3)
C1	H1	0.970	C10	C11	1.47(3)
C1	H2	0.945	C11	C12	1.37(3)
C2	C3	1.46(4)	C11	C16	1.39(3)
C2	H3	0.956	C12	C13	1.37(3)
C2	H4	0.940	C12	H10	0.964
C3	C3A	1.51(3)	C13	C14	1.29(4)
C3	H5	0.952	C13	H11	0.963
C3	H6	0.950	C14	C15	1.41(4)
C3A	O4	1.48(2)	C15	C16	1.40(3)
C3A	C8B	1.59(3)	C15	H12	0.954
C3A	C19	1.54(3)	C16	H13	0.957
O4	C4A	1.37(2)	O17	C18	1.29(3)
C4A	C5	1.44(3)	C18	H14	0.958
C4A	C8A	1.31(2)	C18	H15	0.971
C5	C6	1.42(3)	C18	H16	0.933
C5	H7	0.943	C19	C20	1.41(3)
C6	C7	1.32(3)	C19	C24	1.36(3)
C6	O17	1.41(3)	C20	C21	1.40(3)
C7	C8	1.31(2)	C20	H17	0.945
C7	O9	1.42(2)	C21	C22	1.34(3)
C8	C8A	1.43(2)	C21	H18	0.954
C8	H8	0.955	C22	C23	1.35(3)
C8A	C8B	1.50(2)	C22	O25	1.42(2)

Distances are in angstroms. Estimated standard deviations in the least significant figure are given in parentheses.

Intramolecular Distances (cont)

atom	atom	distance	atom	atom	distance
C23	C24	1.38(3)			
C23	H19	0.947			
C24	H20	0.956			
O25	C26	1.32(3)			
C26	H21	0.954			
C26	H22	0.965			
C26	H23	0.938			

Distances are in angstroms. Estimated standard deviations in the least significant figure are given in parentheses.

Intramolecular Bond Angles

atom	atom	atom	angle	atom	atom	atom	angle
C2	C1	C8B	106(2)	O4	C4A	C8A	116(2)
C2	C1	H1	108.99	C5	C4A	C8A	126(1)
C2	C1	H2	111.08	C4A	C5	C6	111(2)
C8B	C1	H1	110.91	C4A	C5	H7	125.57
C8B	C1	H2	111.94	C6	C5	H7	123.67
H1	C1	H2	108.20	C5	C6	C7	123(2)
C1	C2	C3	105(2)	C5	C6	O17	120(2)
C1	C2	H3	110.13	C7	C6	O17	116(2)
C1	C2	H4	111.04	C6	C7	C8	122(2)
C3	C2	H3	109.57	C6	C7	O9	118(2)
C3	C2	H4	111.15	C8	C7	O9	119(2)
H3	C2	H4	109.81	C7	C8	C8A	121(2)
C2	C3	C3A	101(2)	C7	C8	H8	120.03
C2	C3	H5	113.18	C8A	C8	H8	118.95
C2	C3	H6	112.62	C4A	C8A	C8	115(2)
C3A	C3	H5	110.46	C4A	C8A	C8B	111(1)
C3A	C3	H6	109.58	C8	C8A	C8B	133(2)
H5	C3	H6	109.31	C1	C8B	C3A	102(2)
C3	C3A	O4	110(2)	C1	C8B	C8A	115(2)
C3	C3A	C8B	107(2)	C1	C8B	H9	113.65
C3	C3A	C19	115(1)	C3A	C8B	C8A	100(1)
O4	C3A	C8B	107(1)	C3A	C8B	H9	112.99
O4	C3A	C19	106(2)	C8A	C8B	H9	112.28
C8B	C3A	C19	112(2)	C7	O9	C10	117(2)
C3A	O4	C4A	106(1)	O9	C10	O9A	124(2)
O4	C4A	C5	117(2)	O9	C10	C11	112(2)

Angles are in degrees. Estimated standard deviations in the least significant figure are given in parentheses.

Intramolecular Bond Angles (cont)

atom	atom	atom	angle	atom	atom	atom	angle
O9A	C10	C11	124(3)	C3A	C19	C20	120(2)
C10	C11	C12	121(3)	C3A	C19	C24	125(2)
C10	C11	C16	121(2)	C20	C19	C24	114(2)
C12	C11	C16	117(2)	C19	C20	C21	120(2)
C11	C12	C13	122(3)	C19	C20	H17	122.11
C11	C12	H10	117.75	C21	C20	H17	117.99
C13	C12	H10	119.75	C20	C21	C22	122(2)
C12	C13	C14	118(3)	C20	C21	H18	119.72
C12	C13	H11	121.66	C22	C21	H18	118.41
C14	C13	H11	120.66	C21	C22	C23	120(2)
BR1	C14	C13	122(3)	C21	C22	O25	127(2)
BR1	C14	C15	112(2)	C23	C22	O25	113(2)
C13	C14	C15	126(2)	C22	C23	C24	118(2)
C14	C15	C16	114(3)	C22	C23	H19	120.41
C14	C15	H12	123.63	C24	C23	H19	121.25
C16	C15	H12	122.64	C19	C24	C23	126(2)
C11	C16	C15	122(2)	C19	C24	H20	116.24
C11	C16	H13	118.36	C23	C24	H20	118.05
C15	C16	H13	119.50	C22	O25	C26	115(2)
C6	O17	C18	117(2)	O25	C26	H21	109.45
O17	C18	H14	109.75	O25	C26	H22	108.70
O17	C18	H15	109.63	O25	C26	H23	111.23
O17	C18	H16	110.89	H21	C26	H22	107.95
H14	C18	H15	107.09	H21	C26	H23	110.18
H14	C18	H16	110.24	H22	C26	H23	109.25
H15	C18	H16	109.15				

Angles are in degrees. Estimated standard deviations in the least significant figure are given in parentheses.

Torsion or Conformation Angles

(1)	(2)	(3)	(4)	angle	(1)	(2)	(3)	(4)	angle
BR1	C14	C13	C12	179(2)	O4	C3A	C19	C24	-170(2)
BR1	C14	C15	C16	180(2)	O4	C4A	C5	C6	178(2)
C1	C2	C3	C3A	-43(3)	O4	C4A	C8A	C8	178(2)
C1	C8B	C3A	C3	-2(2)	O4	C4A	C8A	C8B	-3(3)
C1	C8B	C3A	O4	116(2)	C4A	O4	C3A	C8B	1(3)
C1	C8B	C3A	C19	-128(2)	C4A	O4	C3A	C19	-119(2)
C1	C8B	C8A	C4A	-105(3)	C4A	C5	C6	C7	11(4)
C1	C8B	C8A	C8	74(3)	C4A	C5	C6	O17	177(2)
C2	C1	C8B	C3A	-24(2)	C4A	C8A	C8	C7	-2(4)
C2	C1	C8B	C8A	83(2)	C5	C4A	C8A	C8	9(4)
C2	C3	C3A	O4	-89(2)	C5	C4A	C8A	C8B	-171(2)
C2	C3	C3A	C8B	27(3)	C5	C6	C7	C8	-6(5)
C2	C3	C3A	C19	151(2)	C5	C6	C7	O9	-179(3)
C3	C2	C1	C8B	44(3)	C5	C6	O17	C18	6(4)
C3	C3A	O4	C4A	117(2)	C6	C5	C4A	C8A	-14(4)
C3	C3A	C8B	C8A	-120(2)	C6	C7	C8	C8A	0(5)
C3	C3A	C19	C20	126(2)	C6	C7	O9	C10	-92(3)
C3	C3A	C19	C24	-48(3)	C7	C6	O17	C18	173(3)
C3A	O4	C4A	C5	171(2)	C7	C8	C8A	C8B	179(3)
C3A	O4	C4A	C8A	1(3)	C7	O9	C10	O9A	-15(3)
C3A	C8B	C8A	C4A	3(3)	C7	O9	C10	C11	170(2)
C3A	C8B	C8A	C8	-178(3)	C8	C7	C6	O17	-172(3)
C3A	C19	C20	C21	-175(2)	C8	C7	O9	C10	95(3)
C3A	C19	C24	C23	175(2)	C8A	C8	C7	O9	173(2)
O4	C3A	C8B	C8A	-2(2)	C8A	C8B	C3A	C19	113(2)
O4	C3A	C19	C20	4(2)	C8B	C3A	C19	C20	-112(2)

The sign is positive if when looking from atom 2 to atom 3 a clockwise motion of atom 1 would superimpose it on atom 4.

Torsion or Conformation Angles (cont.)

(1)	(2)	(3)	(4)	angle	(1)	(2)	(3)	(4)	angle
C8B	C3A	C19	C24	74(3)					
O9	C7	C6	O17	14(4)					
O9	C10	C11	C12	-176(2)					
O9	C10	C11	C16	-1(3)					
O9A	C10	C11	C12	9(4)					
O9A	C10	C11	C16	-176(2)					
C10	C11	C12	C13	-179(3)					
C10	C11	C16	C15	179(2)					
C11	C12	C13	C14	-8(5)					
C11	C16	C15	C14	8(4)					
C12	C11	C16	C15	-6(4)					
C12	C13	C14	C15	11(6)					
C13	C12	C11	C16	6(4)					
C13	C14	C15	C16	-11(5)					
C19	C20	C21	C22	1(3)					
C19	C24	C23	C22	-1(4)					
C20	C19	C24	C23	0(3)					
C20	C21	C22	C23	-1(3)					
C20	C21	C22	O25	-179(2)					
C21	C20	C19	C24	0(3)					
C21	C22	C23	C24	1(3)					
C21	C22	O25	C26	-4(3)					
C23	C22	O25	C26	178(2)					
C24	C23	C22	O25	179(2)					

The sign is positive if when looking from atom 2 to atom 3 a clockwise motion of atom 1 would superimpose it on atom 4.