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THE CRYSTAL AND MOLECULAR STRUCTURE OF *TRANS*-4-TERT-BUTYLCYCLOHEXYL *p*-TOLUENE SULFONATE

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Abstract—Two independent X-ray analyses of *trans*-4-tert butylcyclohexyl-*p*-toluene sulfonate, $C_{17}H_{26}O_3S$, provide the first detailed structural information on this important class of compounds. The crystals are monoclinic, with average cell dimensions, $a = 11{\cdot}994$, $b = 10{\cdot}475$, $c = 14{\cdot}652$ Å, $\beta = 107{\cdot}62°$, $Z = 4$, the space group being $P2_1/c$. Both determinations of the structure were by direct methods on data collected on single-crystal diffractometers using CuKα radiation. The structures obtained agree within the limits of error but disagree with some of the predictions for this type of system by computer calculations. The predicted twist of the *t*-butyl group was not observed.

INTRODUCTION

A WIDELY ACCEPTED method of locking the mobile cyclohexane system into one of two chair conformations has been the use of the t-butyl group.[1] Any comparisons of data on cyclohexane systems locked into two different conformations rely on assumptions that the cyclohexane ring is unaltered by the different substitutions. This has been criticized because of various experiments showing discrepancies.[2-4]

Recently, Altona and Sundaralingam carried out computer calculations to obtain a quantitative description of the conformation of substituted cyclohexanes.[5] A remarkable feature of the results for t-butylcyclohexanes is the large angle of twist of the t-butyl group accompanied by an asymmetrical distortion of the ring. No experimental diffraction studies on this class of compounds have been reported in the literature, so this current work will provide a test of these predictions. The following paper on the *cis* isomer of this compound will present the comparison of the ring flattening.

It was discovered that a duplication of effort for both compounds had been made when a summary of the University of Arizona (UA) results was published in abstract form.[6] The same crystal structures had been determined at the University of New South Wales (UNSW). Because small changes in bond lengths and angles are being discussed, it was felt that the results of both studies should be published in order to ascertain if the bond length and angle differences between the *cis* and *trans* compounds are meaningful.

DISCUSSION

Values for the bond lengths are in Fig. 1 and the bond angles in Table 1. The arrangement of the atoms about the sulfur atom is approximately tetrahedral. The large

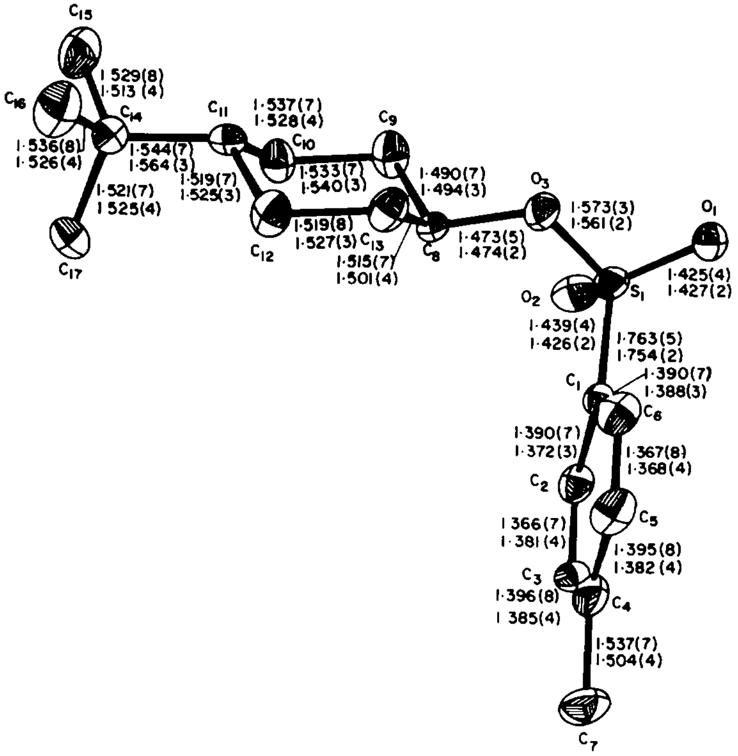

FIG. 1. Bond lengths involving non-hydrogen atoms for *trans*-4-tert butylcyclohexyl-*p*-toluene sulfonate with estimated standard deviations in parentheses. The bond lengths on the top of each pair of numbers are from UA, while the lower numbers are from UNSW.

angle, $O_1—S—O_2$, coupled with the decrease in the angles $C_1—S—O_3$ and $O_1—S—O_3$ would tend to produce minimum hindrance similar to that described by Bartell.[7] The bonds linking sulfur with O_1 and O_2 have almost identical lengths of 1·43 Å. Chemically, the shortness of the bonds suggests that there must be multiple bond character, the most likely explanation for which is that the empty dπ orbitals of the sulfur atom accept electrons *via* overlap from the pπ orbitals of the oxygens, thus giving additional bonding.[8]

The conformation of the sulfonate group is markedly different from that found in *p*-TsOH.[9] The torsional angles about the $C_1—S$ bond are in Table 2. The $S—O_2$ bonds make angles of −12·0° and 171·2° with the $C_1—C_2$ and the $C_1—C_6$ bonds in contrast to being approximately normal in other similar reported compounds.[9] The $S—O_3$ and the $S—O_1$ bonds make angles of about 55° and −56° with the benzene bonds closest to them.

The average value for the bonds of the benzene ring has been calculated as 1·382 Å which is not significantly different from the accepted value for benzene. O'Connell *et al.*[10] have shown theoretically that for a hydrogen substituent in a benzene ring, the carbon and hydrogen atoms are shifted radially towards the ring center, the shift

TABLE 1. BOND ANGLES BETWEEN NON-HYDROGEN ATOMS FOR *trans*-4-tert-BUTYLCYCLOHEXYL TOSYLATE

Atoms	Angle (UNSW)	Angle (UA)
O_1—S—O_2	119·5(1)	119·5(2)
O_1—S—O_3	103·5(1)	104·0(2)
O_2—S—O_3	110·1(1)	109·4(2)
O_1—S—C_1	110·0(1)	110·5(2)
O_2—S—C_1	108·5(1)	108·5(2)
O_3—S—C_1	104·0(1)	103·8(2)
S—C_1—C_2	119·6(2)	120·1(4)
S—C_1—C_6	119·7(2)	120·0(4)
C_1—C_2—C_3	119·5(3)	119·7(5)
C_2—C_3—C_4	121·0(3)	122·3(6)
C_3—C_4—C_5	117·9(3)	116·2(6)
C_4—C_5—C_6	122·3(3)	122·9(6)
C_5—C_6—C_1	118·6(3)	119·0(5)
C_6—C_1—C_2	120·7(3)	119·8(5)
C_3—C_4—C_7	120·4(4)	122·1(6)
C_5—C_4—C_7	121·7(4)	121·7(6)
S—O_3—C_8	120·1(1)	119·7(3)
O_3—C_8—C_9	109·0(2)	109·2(4)
O_3—C_8—C_{13}	106·5(2)	106·2(4)
C_8—C_9—C_{10}	109·8(2)	110·6(5)
C_9—C_{10}—C_{11}	111·8(2)	111·8(5)
C_{10}—C_{11}—C_{12}	108·8(2)	108·6(5)
C_{11}—C_{12}—C_{13}	111·2(2)	113·4(5)
C_{12}—C_{13}—C_8	110·6(2)	110·5(5)
C_{13}—C_8—C_9	112·5(2)	112·1(5)
C_{10}—C_{11}—C_{14}	113·7(2)	113·9(5)
C_{12}—C_{11}—C_{14}	114·6(2)	114·7(5)
C_{11}—C_{14}—C_{15}	110·3(2)	109·8(5)
C_{11}—C_{14}—C_{16}	109·8(2)	110·7(5)
C_{11}—C_{14}—C_{17}	111·2(2)	112·3(5)
C_{15}—C_{14}—C_{16}	108·8(2)	107·5(5)
C_{15}—C_{14}—C_{17}	109·8(2)	109·7(6)
C_{16}—C_{14}—C_{17}	108·2(2)	106·8(5)

shortening the bonds by 0·007 Å. This would account for the low value of the angle at C_4 and the difference in the bond lengths obtained since the bonds C_2—C_3 and C_5—C_6 should be decreased by the full value and the other bonds by half the amount. The mean bond length for the C_2—C_3 and C_5—C_6 bonds is 1·371 Å while the mean bond length for the remaining C—C bonds in the benzene ring is 1·383 Å.

The cyclohexane ring is in the chair form with a small degree of flattening relative to an ideal chair where the valency angles would be tetrahedral and the torsional angles would be 60°. The average torsional angle found is 55·9° which is identical to the recent electron diffraction study of cyclohexane by Buys and Geise.[11] The average valency angle is 111·0° which also is equal to the value found in this most recent study. The average C—C bond for the chair is 1·519° Å but there are significant deviations from

TABLE 2. TORSIONAL ANGLES IN *trans*-4-tert BUTYLCYCLOHEXYL-*p*-TOLUENE SULFONATE FROM UA DATA WITH STANDARD DEVIATIONS IN PARENTHESES

Atoms	Torsional Angle (degrees)
O_2—S—C_1—C_2	−12·0(5)
O_2—S—C_1—C_6	171·2(4)
O_3—S—C_1—C_2	−128·3(4)
O_3—S—C_1—C_6	54·9(5)
O_1—S—C_1—C_2	120·8(4)
O_1—S—C_1—C_6	−56·0(5)
C_8—C_9—C_{10}—C_{11}	−57·1(7)
C_9—C_{10}—C_{11}—C_{12}	55·3(6)
C_{10}—C_{11}—C_{12}—C_{13}	−54·9(7)
C_{11}—C_{12}—C_{13}—C_8	55·0(7)
C_{12}—C_{13}—C_8—C_9	−54·9(7)
C_{13}—C_8—C_9—C_{10}	56·4(7)
C_{10}—C_{11}—C_{14}—C_{15}	−60·1(7)
C_{10}—C_{11}—C_{14}—C_{16}	178·6(5)
C_{10}—C_{11}—C_{14}—C_{17}	−62·2(7)
C_{12}—C_{11}—C_{14}—C_{15}	−173·8(5)
C_{12}—C_{11}—C_{14}—C_{16}	−55·3(7)
C_{12}—C_{11}—C_{14}—C_{17}	63·8(7)

TABLE 3. ATOMIC COORDINATES FOR *trans*-t-tert-BUTYLCYCLOHEXYL *p*-TOLUENE SULFONATE WITH STANDARD DEVIATIONS IN PARENTHESES

	UA			UNSW		
Atoms	*x/a*	*y/b*	*z/c*	*x/a*	*y/b*	*z/c*
S	0·00630(12)	0·06046(14)	0·71056(10)	0·00604(4)	0·06063(6)	0·71035(4)
O_1	0·1243(3)	0·0273(4)	0·7601(3)	0·1241(1)	0·0269(2)	0·7604(1)
O_2	−0·0173(3)	0·1675(3)	0·6461(3)	−0·0167(1)	0·1674(2)	0·6469(1)
O_3	−0·0456(3)	−0·0641(3)	0·6538(2)	−0·0452(1)	−0·0636(1)	0·6543(1)
C_1	−0·0749(4)	0·0791(5)	0·7918(4)	−0·0743(2)	0·0795(2)	0·7916(1)
C_2	−0·1288(5)	0·1948(5)	0·7977(4)	−0·1275(2)	0·1943(3)	0·7966(2)
C_3	−0·1861(5)	0·2108(6)	0·8645(4)	−0·1858(2)	0·2109(3)	0·8640(2)
C_4	−0·1927(5)	0·1142(7)	0·9282(4)	−0·1915(2)	0·1137(3)	0·9265(2)
C_5	−0·1385(6)	−0·0009(6)	0·9193(4)	−0·1379(2)	−0·0009(3)	0·9188(2)
C_6	−0·0803(5)	−0·0198(5)	0·8534(4)	−0·0796(2)	−0·0200(3)	0·8528(2)
C_7	−0·2569(6)	0·1326(7)	1·0034(4)	−0·2541(4)	0·1338(6)	1·0001(3)
C_8	−0·1661(4)	−0·0636(5)	0·5885(4)	−0·1656(2)	−0·0647(2)	0·5884(1)
C_9	−0·1629(5)	−0·0481(6)	0·4883(4)	−0·1617(2)	−0·0479(3)	0·4882(2)
C_{10}	−0·2857(5)	−0·0619(6)	0·4173(4)	−0·2856(2)	−0·0623(3)	0·4175(2)
C_{11}	−0·3413(4)	−0·1908(5)	0·4284(4)	−0·3401(2)	−0·1912(2)	0·4283(1)
C_{12}	−0·3403(5)	−0·2050(6)	0·5317(4)	−0·3403(2)	−0·2057(3)	0·5319(2)
C_{13}	−0·2197(6)	−0·1898(6)	0·6034(4)	−0·2182(2)	−0·1903(3)	0·6030(2)
C_{14}	−0·4612(5)	−0·2142(6)	0·3534(4)	−0·4611(2)	−0·2157(3)	0·3517(2)
C_{15}	−0·4482(6)	−0·2116(7)	0·2529(5)	−0·4489(3)	−0·2097(5)	0·2519(3)

TABLE 3—*cont.*

	UA			UNSW		
Atom	*x/a*	*y/b*	*z/c*	*x/a*	*y/b*	*z/c*
C_{16}	−0·5092(6)	−0·3460(7)	0·3678(5)	−0·5067(3)	−0·3476(4)	0·3669(3)
C_{17}	−0·5521(5)	−0·1167(7)	0·3604(5)	−0·5514(3)	−0·1168(4)	0·3590(3)
H_1	−0·132(3)	0·250(4)	0·752(3)	−0·123(2)	0·255(3)	0·750(2)
H_2	−0·228(3)	0·287(4)	0·870(3)	−0·226(2)	0·285(3)	0·870(2)
H_3	−0·138(4)	−0·067(4)	0·956(3)	−0·139(3)	−0·066(4)	0·953(2)
H_4	−0·045(4)	−0·089(4)	0·853(3)	−0·049(3)	−0·094(3)	0·849(2)
H_{5A}*				−0·205(7)	0·155(12)	1·058(7)
H_{6A}*				−0·329(6)	0·210(9)	0·976(6)
H_{7A}*				−0·269(8)	0·055(10)	1·004(7)
H_{5B}*				−0·214(6)	0·050(10)	1·055(7)
H_{6B}*				−0·236(7)	0·224(9)	1·040(7)
H_{7B}*				−0·313(7)	0·137(11)	1·020(7)
H_8	−0·210(4)	0·013(4)	0·607(3)	−0·206(2)	−0·002(3)	0·601(2)
H_9	−0·123(4)	0·039(5)	0·481(3)	−0·123(2)	0·044(3)	0·479(2)
H_{10}	−0·105(4)	0·111(5)	0·479(4)	−0·114(2)	0·125(3)	0·480(2)
H_{11}	−0·283(4)	−0·052(4)	0·344(3)	−0·290(2)	−0·060(3)	0·353(2)
H_{12}	−0·345(4)	0·009(5)	0·436(3)	−0·336(2)	0·005(3)	0·428(2)
H_{13}	−0·296(4)	−0·245(5)	0·422(3)	−0·285(2)	−0·252(4)	0·418(2)
H_{14}	−0·408(4)	−0·140(4)	0·546(3)	−0·396(2)	−0·144(3)	0·542(2)
H_{15}	−0·377(4)	−0·287(5)	0·541(3)	−0·370(2)	−0·291(3)	0·542(2)
H_{16}	−0·221(4)	−0·193(5)	0·668(3)	−0·223(2)	−0·201(3)	0·667(2)
H_{17}	−0·171(4)	−0·252(5)	0·584(3)	−0·165(3)	−0·255(3)	0·593(2)
H_{18}	−0·393(5)	−0·246(5)	0·248(4)	−0·391(3)	−0·283(5)	0·254(2)
H_{19}	−0·430(5)	−0·137(5)	0·243(4)	−0·413(3)	−0·140(4)	0·236(3)
H_{20}	−0·516(5)	−0·222(5)	0·203(4)	−0·521(3)	−0·232(4)	0·208(2)
H_{21}	−0·576(5)	−0·348(5)	0·314(4)	−0·573(3)	−0·360(4)	0·317(3)
H_{22}	−0·455(5)	−0·394(6)	0·359(4)	−0·436(3)	−0·407(4)	0·357(3)
H_{23}	−0·505(4)	−0·382(5)	0·431(4)	−0·521(3)	−0·357(4)	0·429(3)
H_{24}	−0·627(5)	−0·137(5)	0·303(4)	−0·622(3)	−0·131(4)	0·311(3)
H_{25}	−0·568(5)	−0·099(5)	0·418(4)	−0·567(3)	−0·107(4)	0·420(3)
H_{26}	−0·522(6)	−0·058(6)	0·334(5)	−0·531(3)	−0·036(5)	0·352(3)

* "$\frac{1}{2}$ hydrogen".

this average value. The bonds C_8—C_9 and C_8—C_{13} are short and are considered to be a result of sp hybrid bonding with oxygen.

The bond length C_{11}—C_{14} increases to 1·554 Å which results most probably from a gauche interaction between non-bonded neighbors. Further evidence of strain in the t-butyl portion of the molecule can be found in the average bond angle within the group (108·5°). In a similar way, the bond angles C_{10}, C_{11}, C_{14} and C_{12}, C_{11}, C_{14} increase to 114·2°. From Table 2, the torsional angles about the C_{11}—C_{14} bond show no twist of the nature predicted by Altona and Sundaralingam.[5] No evidence of disorder was found in the difference map and the thermal parameters for the t-butyl group are not anomalously high.

An intermolecular scan of bond lengths shows no anomalously short intermolecular contacts.

Table 4. Thermal parameters from UA and UNSW with standard deviations in parentheses. For each atom, the first line is the UA value and the second the UNSW value. Temperature factors are of form $\exp[-(\beta_{11}h^2 + \beta_{22}k^2 + \beta_{33}l^2 + 2\beta_{12}hk + 2\beta_{13}hl + 2\beta_{23}kl]$.

Atom	β_{11}	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}
S	0·00830(13)	0·00892(16)	0·00576(9)	0·00114(12)	0·00238(9)	−0·00150(10)
	0·00961(4)	0·01104(6)	0·00712(3)	−0·00110(4)	0·00283(3)	−0·00148(4)
O_1	0·0077(3)	0·0153(5)	0·0082(3)	−0·0013(3)	0·0021(3)	−0·0035(3)
	0·0083(1)	0·0184(3)	0·0098(1)	−0·0009(1)	0·0020(1)	−0·0035(1)
O_2	0·0154(5)	0·0099(4)	0·0081(3)	−0·0018(4)	0·0057(3)	−0·0007(3)
	0·0166(2)	0·0111(2)	0·0090(1)	−0·0013(2)	0·0059(1)	0·0003(1)
O_3	0·0086(3)	0·0087(4)	0·0057(2)	0·0006(3)	0·0007(2)	−0·0015(3)
	0·0090(1)	0·0099(2)	0·0067(1)	0·0008(1)	0·0010(1)	−0·0015(1)
C_1	0·0079(5)	0·0080(6)	0·0053(3)	−0·0012(4)	0·0020(3)	−0·0012(4)
	0·0086(1)	0·0095(2)	0·0063(1)	−0·0013(1)	0·0017(1)	−0·0012(1)
C_2	0·0090(5)	0·0087(6)	0·0059(4)	−0·0007(5)	0·0018(4)	−0·0002(4)
	0·0099(2)	0·0098(3)	0·0073(1)	−0·0006(2)	0·0019(1)	−0·0011(1)
C_3	0·0090(6)	0·0118(8)	0·0082(5)	0·0003(5)	0·0026(4)	−0·0038(5)
	0·0105(2)	0·0137(3)	0·0080(2)	−0·0004(2)	0·0023(1)	−0·0028(2)
C_4	0·0093(6)	0·0149(9)	0·0063(4)	−0·0033(6)	0·0016(4)	−0·0026(5)
	0·0101(2)	0·0170(4)	0·0068(1)	−0·0033(2)	0·0023(1)	−0·0028(2)
C_5	0·0131(7)	0·0138(8)	0·0058(4)	−0·0039(6)	0·0030(4)	0·0010(5)
	0·0135(2)	0·0149(4)	0·0072(2)	−0·0038(2)	0·0029(1)	0·0002(2)
C_6	0·0119(6)	0·0087(6)	0·0070(4)	−0·0000(5)	0·0023(4)	0·0003(4)
	0·0128(2)	0·0107(3)	0·0075(2)	−0·0010(2)	0·0027(1)	0·0000(2)
C_7	0·0147(7)	0·0268(12)	0·0076(5)	−0·0037(8)	0·0068(5)	−0·0045(6)
	0·0154(4)	0·0307(9)	0·0088(2)	−0·0042(5)	0·0059(2)	−0·0049(4)
C_8	0·0072(5)	0·0087(6)	0·0052(3)	0·0003(4)	0·0017(3)	−0·0009(4)
	0·0082(1)	0·0100(2)	0·0062(1)	0·0007(2)	0·0020(1)	−0·0007(1)
C_9	0·0085(6)	0·0135(8)	0·0061(4)	−0·0021(5)	0·0018(4)	0·0017(5)
	0·0091(2)	0·0163(4)	0·0064(1)	−0·0019(2)	0·0021(1)	0·0014(2)
C_{10}	0·0087(6)	0·0140(8)	0·0055(4)	−0·0012(5)	0·0015(4)	0·0016(5)
	0·0097(2)	0·0153(4)	0·0064(1)	−0·0011(2)	0·0015(1)	0·0020(2)
C_{11}	0·0079(5)	0·0094(6)	0·0053(3)	0·0010(4)	0·0023(3)	−0·0012(4)
	0·0085(2)	0·0119(3)	0·0060(1)	0·0008(2)	0·0025(1)	−0·0011(1)
C_{12}	0·0104(6)	0·0127(8)	0·0061(4)	−0·0037(6)	0·0031(4)	0·0000(5)
	0·0119(2)	0·0152(4)	0·0067(1)	−0·0036(2)	0·0032(1)	−0·0005(2)
C_{13}	0·0120(7)	0·0119(8)	0·0051(4)	−0·0021(6)	0·0022(4)	−0·0002(5)
	0·0131(2)	0·0136(3)	0·0056(1)	−0·0028(2)	0·0021(1)	0·0005(2)
C_{14}	0·0083(5)	0·0124(7)	0·0061(4)	−0·0003(5)	0·0020(4)	−0·0020(4)
	0·0091(2)	0·0138(3)	0·0073(1)	0·0001(2)	0·0020(1)	−0·0024(2)
C_{15}	0·0142(8)	0·0257(13)	0·0063(4)	−0·0013(8)	0·0008(5)	−0·0027(6)
	0·0142(3)	0·0258(7)	0·0069(2)	−0·0011(3)	0·0011(2)	−0·0023(3)
C_{16}	0·0147(8)	0·0179(10)	0·0086(5)	−0·0048(7)	0·0016(5)	−0·0022(6)
	0·0151(3)	0·0182(5)	0·0107(2)	−0·0047(3)	0·0023(2)	−0·0038(3)
C_{17}	0·0087(6)	0·0215(11)	0·0111(6)	0·0030(7)	0·0011(5)	−0·0051(6)
	0·0093(2)	0·0219(6)	0·0139(3)	0·0027(3)	0·0004(2)	−0·0064(3)

EXPERIMENTAL

The *trans*-4-tert-butylcyclohexyl-*p*-toluene sulfonate crystals were colorless plate-like prisms elongated in the *c* direction. Systematic absences among the reflections indicated that the space group was $P2_1/c$. The unit cell parameters were measured at UNSW to be $a = 11{\cdot}985(5)$, $b = 10{\cdot}466(5)$, $c = 14{\cdot}643(5)$, $\beta = 107{\cdot}640(5)$, while at UA they were found to be $a = 12{\cdot}002(2)$, $b = 10{\cdot}483(2)$, $c = 14{\cdot}661(5)$, $\beta = 107{\cdot}59(3)$. The agreement between the two sets of cell parameters is similar to that found by another study where independent determinations in different laboratories were made on another compound.[12]

The integrated intensities at UNSW were recorded by a θ–2θ scan using the five value method of Hoppe.[13] The scan range varied from 1·00° at low two theta values to 1·74° at high two theta values. A standard reflection, measured after every 15 reflections, revealed no crystal or instrumental drift. A total of 3323 reflections were observed, being approximately 90% of those with $\theta < 70°$, the angular limit of the machine. The intensities were corrected for Lorentz, polarization, and absorption factors.

At UA, a continuous θ–2θ scan was employed with the base width being 1·8°. An increase in the scan was made according to the α_1–α_2 splitting. Ten second background counts were made at the extremes of the scan. There were 2516 observed reflections. The intensities were corrected for Lorentz and polarization factors but not for absorption.

At UNSW, phases were determined directly from the observed intensities using the logical symbolic addition method for the automatic solution of centrosymmetric crystals.[14] All 21 non-hydrogen atoms were located in the E map computed from these phases. The initial R based on these coordinates and an overall temperature factor of 4·6 Å^2 was 29·8%. Refinement with isotropic and anisotropic temperature factors reduced the residual to 9·3%. A high angle refinement was run at this stage to remove any effects of bonding asphericity.

Twenty-three hydrogen atoms were clearly visible in a difference map computed at this stage at UNSW. The other three hydrogens, those attached to C_7, were indicated by two smears extending over approximately $\frac{1}{2}$ of the unit cell in the Y direction and approximately $\frac{1}{2}$ density of a hydrogen atom indicating a distortion producing a limited angular rotation about the axis of torsional oscillation. These smears showed six slight peaks and these were taken as a set of six 'half-hydrogens' with each hydrogen occupying two positions with equal probability. The hydrogens were given constant temperature factors equal to those of the carbon atoms to which they were attached and no attempt was made to determine their individual motions. Their positions were refined along with the positions and thermal parameters of the heavier atoms leading to an R of 4·7%. Tables 3 and 4 list the final fractional coordinates and thermal parameters of the atoms for this determination as well as that done at UA. The thermal parameters for hydrogen atoms were not refined. At UNSW, they were given the same values as the heavier atoms to which they were attached. At UA, they were fixed at the isotropic B values of 5 Å^2 for hydrogens attached to a ring and 8 Å^2 for methyl hydrogens.

At UA, the solution of the structure was by the reiterative application of Sayre's equation as applied by the program written by R. E. Long.[15] All non-hydrogen atoms were found and refinement proceeded smoothly to an R value of 0·107. At this stage, a difference map was examined and all hydrogen atoms with the exception of the ones attached to C^7 were found. No attempt was made to locate a disordered or rotating model to the density remaining around C_7. This was because this methyl group is remote from the area of interest, that of the cyclohexane portion. For these latter stages of refinement, the molecule was refined in two parts. The alternate parts consisted of the methyl benzene and the t-butyl cyclohexane groups with the SO_3 group being common to both parts. The final R value was 0·080 with the scattering form factors being those of Hanson, Herman, Lea, and Skillman.[11]

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