

X-Ray Structure Analysis to Determine the Stereochemistry of 1a. The less soluble diastereomeric salt of racemic **1a** with (*R*)- α -methyl benzylamine (**4**, Figure 1) was crystallized from ethyl acetate as monoclinic crystals with the chiral space group $P2_1$. The molecular structure obtained from the X-ray analysis is identical to that shown in Figure 1. By considering *R*-configuration for α -methyl benzylamine, the absolute configuration of the indane acid was determined to be *1S*, *10S*. Figure 2 is an ORTEP representation of the salt with the atomic numbering of non-hydrogen atoms.

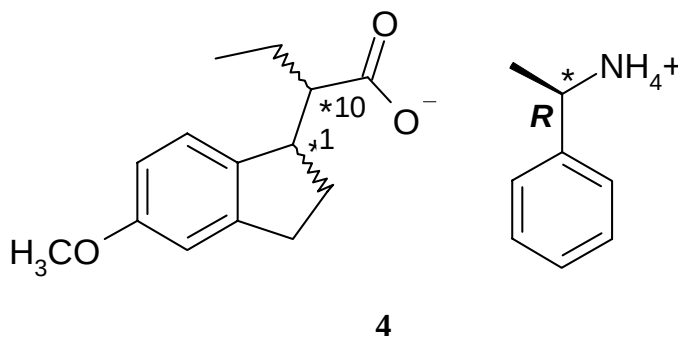


Figure 1. Chemical structure of the less soluble diastereomeric salt of **1a** with (*R*)- α -methyl benzylamine (**4**).

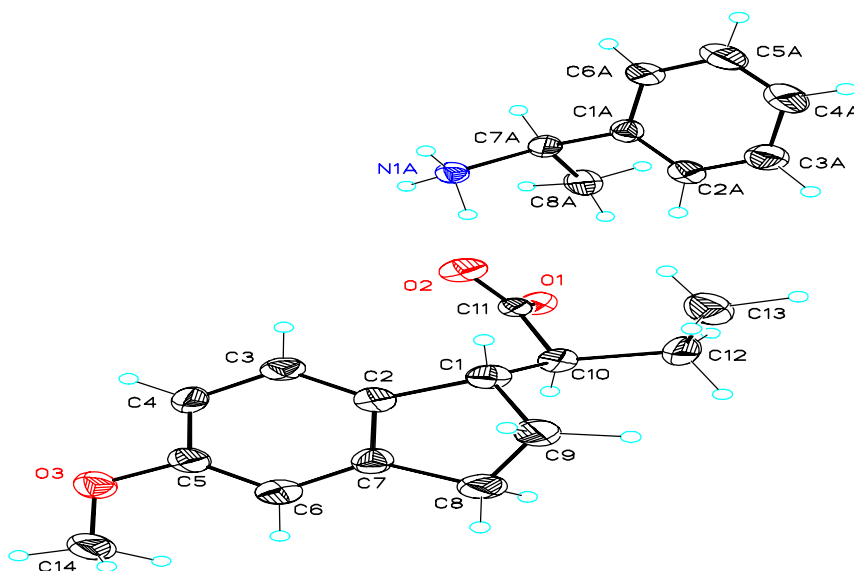


Figure 2. ORTEP representation of **4** with the atomic numbering of non-hydrogen atoms.

Table 1. Crystal data and structure refinement for **4**.

Identification code	xma001ag
Empirical formula	C ₂₂ H ₂₉ N ₁ O ₃
Formula weight	236.97
Temperature	153(2) K
Wavelength	0.71073 Å
Crystal system	Monoclinic
Space group	<i>P</i> 2 ₁
Unit cell dimensions	<i>a</i> = 12.3181(9) Å α = 90°. <i>b</i> = 5.7793(4) Å β = 107.951(2)°. <i>c</i> = 14.4959(11) Å γ = 90°.
Volume	981.73(12) Å ³
<i>Z</i>	3
Density (calculated)	1.202 Mg/m ³
Absorption coefficient	0.079 mm ⁻¹
<i>F</i> (000)	384
Crystal size	0.01 x 0.04 x 0.4 mm ³
Theta range for data collection	1.48 to 27.64°.
Index ranges	-16 ≤ <i>h</i> ≤ 16, -7 ≤ <i>k</i> ≤ 7, -18 ≤ <i>l</i> ≤ 18
Reflections collected	11910
Independent reflections	4465 [<i>R</i> (int) = 0.0569]
Completeness to theta = 27.64°	98.2 %
Absorption correction	SADABS (Bruker-AXS)
Refinement method	Full-matrix least-squares on <i>F</i> ²
Data / restraints / parameters	4465 / 1 / 351
Goodness-of-fit on <i>F</i> ²	0.886
Final <i>R</i> indices [<i>I</i> > 2σ(<i>I</i>)]	<i>R</i> 1 = 0.0452, <i>wR</i> 2 = 0.0875
<i>R</i> indices (all data)	<i>R</i> 1 = 0.0922, <i>wR</i> 2 = 0.1013
Absolute structure parameter	-2.4(12)
Largest diff. peak and hole	0.195 and -0.184 e.Å ⁻³

Table 2. Bond lengths [Å] for **4**.

O(1)-C(11)	1.282(3)
N(1A)-C(7A)	1.507(3)
C(1)-C(2)	1.525(3)
C(1)-C(10)	1.544(3)
C(1)-C(9)	1.552(4)
C(1A)-C(2A)	1.383(4)
C(1A)-C(6A)	1.388(4)
C(1A)-C(7A)	1.517(3)
O(2)-C(11)	1.239(3)
C(2)-C(7)	1.385(4)
C(2)-C(3)	1.389(4)
C(2A)-C(3A)	1.396(4)
O(3)-C(5)	1.383(3)
O(3)-C(14)	1.427(4)
C(3)-C(4)	1.396(3)
C(3A)-C(4A)	1.385(4)
C(4)-C(5)	1.394(4)
C(4A)-C(5A)	1.368(4)
C(5)-C(6)	1.386(4)
C(5A)-C(6A)	1.394(4)
C(6)-C(7)	1.392(4)
C(7A)-C(8A)	1.510(4)
C(7)-C(8)	1.506(4)
C(8)-C(9)	1.525(4)
C(10)-C(11)	1.520(3)
C(10)-C(12)	1.552(3)
C(12)-C(13)	1.517(4)

Table 3. Bond angles [°] for **4**.

C(2)-C(1)-C(10)	111.0(2)
C(2)-C(1)-C(9)	101.1(2)
C(10)-C(1)-C(9)	113.4(2)
C(2A)-C(1A)-C(6A)	118.9(2)
C(2A)-C(1A)-C(7A)	121.3(2)
C(6A)-C(1A)-C(7A)	119.9(2)
C(7)-C(2)-C(3)	119.7(2)
C(7)-C(2)-C(1)	110.4(2)
C(3)-C(2)-C(1)	129.9(2)
C(1A)-C(2A)-C(3A)	120.4(3)
C(5)-O(3)-C(14)	116.5(2)
C(2)-C(3)-C(4)	119.3(3)
C(4A)-C(3A)-C(2A)	120.2(3)
C(5)-C(4)-C(3)	120.2(2)
C(5A)-C(4A)-C(3A)	119.5(3)
O(3)-C(5)-C(6)	123.8(2)
O(3)-C(5)-C(4)	115.4(2)
C(6)-C(5)-C(4)	120.8(2)
C(4A)-C(5A)-C(6A)	120.6(3)
C(5)-C(6)-C(7)	118.2(3)
C(1A)-C(6A)-C(5A)	120.4(3)
N(1A)-C(7A)-C(8A)	110.0(2)
N(1A)-C(7A)-C(1A)	108.96(19)
C(8A)-C(7A)-C(1A)	114.3(2)
C(2)-C(7)-C(6)	121.8(2)
C(2)-C(7)-C(8)	110.6(2)
C(6)-C(7)-C(8)	127.6(3)
C(7)-C(8)-C(9)	102.9(2)
C(8)-C(9)-C(1)	105.4(2)
C(11)-C(10)-C(1)	112.1(2)
C(11)-C(10)-C(12)	107.7(2)
C(1)-C(10)-C(12)	113.9(2)
O(2)-C(11)-O(1)	122.9(2)
O(2)-C(11)-C(10)	120.8(2)
O(1)-C(11)-C(10)	116.3(2)
C(13)-C(12)-C(10)	113.5(2)

Table 4. Torsion angles [°] for **4**.

C(10)-C(1)-C(2)-C(7)	100.5(3)
C(9)-C(1)-C(2)-C(7)	-20.1(3)
C(10)-C(1)-C(2)-C(3)	-79.6(3)
C(9)-C(1)-C(2)-C(3)	159.8(3)
C(6A)-C(1A)-C(2A)-C(3A)	0.6(4)
C(7A)-C(1A)-C(2A)-C(3A)	-178.3(2)
C(7)-C(2)-C(3)-C(4)	0.3(4)
C(1)-C(2)-C(3)-C(4)	-179.6(2)
C(1A)-C(2A)-C(3A)-C(4A)	-0.5(4)
C(2)-C(3)-C(4)-C(5)	-1.2(4)
C(2A)-C(3A)-C(4A)-C(5A)	0.4(4)
C(14)-O(3)-C(5)-C(6)	-0.1(4)
C(14)-O(3)-C(5)-C(4)	-179.0(2)
C(3)-C(4)-C(5)-O(3)	-179.5(2)
C(3)-C(4)-C(5)-C(6)	1.5(4)
C(3A)-C(4A)-C(5A)-C(6A)	-0.3(4)
O(3)-C(5)-C(6)-C(7)	-179.7(2)
C(4)-C(5)-C(6)-C(7)	-0.9(4)
C(2A)-C(1A)-C(6A)-C(5A)	-0.6(4)
C(7A)-C(1A)-C(6A)-C(5A)	178.4(2)
C(4A)-C(5A)-C(6A)-C(1A)	0.4(4)
C(2A)-C(1A)-C(7A)-N(1A)	70.9(3)
C(6A)-C(1A)-C(7A)-N(1A)	-108.1(2)
C(2A)-C(1A)-C(7A)-C(8A)	-52.6(3)
C(6A)-C(1A)-C(7A)-C(8A)	128.4(3)
C(3)-C(2)-C(7)-C(6)	0.4(4)
C(1)-C(2)-C(7)-C(6)	-179.7(2)
C(3)-C(2)-C(7)-C(8)	-177.7(2)
C(1)-C(2)-C(7)-C(8)	2.2(3)
C(5)-C(6)-C(7)-C(2)	-0.1(4)
C(5)-C(6)-C(7)-C(8)	177.6(3)
C(2)-C(7)-C(8)-C(9)	17.0(3)
C(6)-C(7)-C(8)-C(9)	-160.9(3)
C(7)-C(8)-C(9)-C(1)	-29.1(3)
C(2)-C(1)-C(9)-C(8)	29.9(3)
C(10)-C(1)-C(9)-C(8)	-89.0(3)
C(2)-C(1)-C(10)-C(11)	71.2(3)
C(9)-C(1)-C(10)-C(11)	-175.8(2)
C(2)-C(1)-C(10)-C(12)	-166.1(2)
C(9)-C(1)-C(10)-C(12)	-53.1(3)
C(1)-C(10)-C(11)-O(2)	25.1(3)
C(12)-C(10)-C(11)-O(2)	-101.0(3)
C(1)-C(10)-C(11)-O(1)	-156.4(2)
C(12)-C(10)-C(11)-O(1)	77.6(2)
C(11)-C(10)-C(12)-C(13)	74.7(3)
C(1)-C(10)-C(12)-C(13)	-50.3(3)