

***trans*-(*N*-*tert*-Butoxycarbonyl)-1-amino-1-carboxyl-2-(2,2-diphenylethenyl)cyclopropane**

24.

The cyclopropane crystallises from chloroform/pentane by vapour diffusion to give single crystals; Crystallographic data are presented in Tables 1 and 2. A single crystal of **24** was coated in high-vacuum grease and mounted on a glass fibre. X-ray measurements were made using a Bruker SMART CCD area-detector diffractometer with Mo-K α radiation ($\lambda = 0.71073 \text{ \AA}$).¹ Intensities were integrated² from several series of exposures, each exposure covering 0.3° in θ , and the total data set being a hemisphere. Absorption corrections were applied, based on multiple and symmetry-equivalent measurements.³ The structure was solved by direct methods and refined by least squares on weighted F^2 values for all reflections.⁴ All non-hydrogen atoms were assigned anisotropic displacement parameters and refined without positional constraints. All hydrogen atoms were constrained to ideal geometries and refined with fixed isotropic displacement parameters 1.2 times that of their parent atoms. Complex neutral-atom scattering factors were used.⁵

Table 1. Crystal data and structure refinement for *trans*-(*N*-*tert*-Butoxycarbonyl)-1-amino-1-carboxyl-2-(2,2-diphenylethenyl)cyclopropane **24**.

Identification code	LAA-24
Empirical formula	C ₂₄ H ₂₆ Cl ₃ N O ₄ (Co crystallises with CHCl ₃)
Formula weight	498.81
Temperature	173(2) K
Wavelength	0.71073 \AA
Crystal system	Monoclinic
Space group	P2(1)/c
Unit cell dimensions	$a = 12.425(3) \text{ \AA}$ $\beta = 90^\circ$ $b = 18.223(5) \text{ \AA}$ $\beta = 103.042(17)^\circ$ $c = 11.601(2) \text{ \AA}$ $\beta = 90^\circ$
Volume	2558.9(11) \AA^3
Z	4
Density (calculated)	1.295 Mg/m ³
Absorption coefficient	0.387 mm ⁻¹
F(000)	1040
Crystal size	0.2 x 0.2 x 0.05 mm
θ range for data collection	2.02 to 24.01 $^\circ$
Index ranges	-14 $\leq h \leq$ 9, -20 $\leq k \leq$ 20, -13 $\leq l \leq$ 12
Reflections collected	12277
Independent reflections	4000 [$R_{\text{int}} = 0.0381$]
Completeness to $\theta = 24.01^\circ$	99.7 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	0.981 and 0.926
Refinement method	Full-matrix least-squares on F^2

Data / restraints / parameters	4000 / 0 / 295
Goodness-of-fit on F^2	$S = 1.067$
R indices [for 2905 reflections with $I > 2\sigma(I)$]	$R_1 = 0.0745$, $wR_2 = 0.1994$
R indices (for all 4000 data)	$R_1 = 0.0991$, $wR_2 = 0.2216$
Weighting scheme	$w^{-1} = \sigma^2(F_o^2) + (aP)^2$, where $P = [\max(F_o^2, 0) + 2F_c^2]/3$ $a = 0.1057$
Largest diff. peak and hole	1.435 and -1.008 $e\text{\AA}^{-3}$

Table 2 Bond lengths [\AA] and angles [$^\circ$] for *trans*-(*N*-*tert*-Butoxycarbonyl)-1-amino-1-carboxyl-2-(2,2-diphenylethenyl)cyclopropane **24**.

C(1)-C(2)	1.383(5)	C(15)-C(17)	1.538(5)
C(1)-C(6)	1.389(5)	C(15)-H(15)	1.0000
C(1)-H(1)	0.9500	C(16)-C(17)	1.505(5)
C(2)-C(3)	1.382(6)	C(16)-H(16A)	0.9900
C(2)-H(2A)	0.9500	C(16)-H(16B)	0.9900
C(3)-C(4)	1.384(6)	C(17)-N(1)	1.443(5)
C(3)-H(3)	0.9500	C(17)-C(18)	1.499(5)
C(4)-C(5)	1.392(5)	C(18)-O(1)	1.211(4)
C(4)-H(4)	0.9500	C(18)-O(2)	1.321(5)
C(5)-C(6)	1.397(5)	C(19)-O(3)	1.227(5)
C(5)-H(5)	0.9500	C(19)-O(4)	1.332(5)
C(6)-C(7)	1.493(5)	C(19)-N(1)	1.343(5)
C(7)-C(14)	1.338(5)	C(20)-O(4)	1.488(4)
C(7)-C(8)	1.495(5)	C(20)-C(22)	1.509(7)
C(8)-C(13)	1.396(6)	C(20)-C(23)	1.512(6)
C(8)-C(9)	1.398(6)	C(20)-C(21)	1.516(6)
C(9)-C(10)	1.395(6)	C(21)-H(21A)	0.9800
C(9)-H(9)	0.9500	C(21)-H(21B)	0.9800
C(10)-C(11)	1.370(7)	C(21)-H(21C)	0.9800
C(10)-H(10)	0.9500	C(22)-H(22A)	0.9800
C(11)-C(12)	1.375(7)	C(22)-H(22B)	0.9800
C(11)-H(11)	0.9500	C(22)-H(22C)	0.9800
C(12)-C(13)	1.388(6)	C(23)-H(23A)	0.9800
C(12)-H(12)	0.9500	C(23)-H(23B)	0.9800
C(13)-H(13)	0.9500	C(23)-H(23C)	0.9800
C(14)-C(15)	1.487(5)	N(1)-H(1A)	0.9200
C(14)-H(14)	0.9500	O(2)-H(2)	0.69(9)
C(15)-C(16)	1.496(5)	C(24)-Cl(2)	1.704(6)

C(24)-Cl(1)	1.716(7)	C(12)-C(13)-H(13)	119.7
C(24)-Cl(3)	1.737(5)	C(8)-C(13)-H(13)	119.7
C(24)-H(24)	1.0000	C(7)-C(14)-C(15)	126.2(3)
C(2)-C(1)-C(6)	121.3(4)	C(7)-C(14)-H(14)	116.9
C(2)-C(1)-H(1)	119.3	C(15)-C(14)-H(14)	116.9
C(6)-C(1)-H(1)	119.3	C(14)-C(15)-C(16)	119.9(3)
C(3)-C(2)-C(1)	120.2(4)	C(14)-C(15)-C(17)	120.7(3)
C(3)-C(2)-H(2A)	119.9	C(16)-C(15)-C(17)	59.5(2)
C(1)-C(2)-H(2A)	119.9	C(14)-C(15)-H(15)	115.2
C(2)-C(3)-C(4)	119.5(4)	C(16)-C(15)-H(15)	115.2
C(2)-C(3)-H(3)	120.2	C(17)-C(15)-H(15)	115.2
C(4)-C(3)-H(3)	120.2	C(15)-C(16)-C(17)	61.7(2)
C(3)-C(4)-C(5)	120.4(4)	C(15)-C(16)-H(16A)	117.6
C(3)-C(4)-H(4)	119.8	C(17)-C(16)-H(16A)	117.6
C(5)-C(4)-H(4)	119.8	C(15)-C(16)-H(16B)	117.6
C(4)-C(5)-C(6)	120.4(4)	C(17)-C(16)-H(16B)	117.6
C(4)-C(5)-H(5)	119.8	H(16A)-C(16)-H(16B)	114.7
C(6)-C(5)-H(5)	119.8	N(1)-C(17)-C(18)	117.4(3)
C(1)-C(6)-C(5)	118.2(3)	N(1)-C(17)-C(16)	118.6(3)
C(1)-C(6)-C(7)	120.3(3)	C(18)-C(17)-C(16)	117.6(3)
C(5)-C(6)-C(7)	121.4(3)	N(1)-C(17)-C(15)	115.6(3)
C(14)-C(7)-C(6)	120.1(3)	C(18)-C(17)-C(15)	115.4(3)
C(14)-C(7)-C(8)	122.9(3)	C(16)-C(17)-C(15)	58.9(2)
C(6)-C(7)-C(8)	116.9(3)	O(1)-C(18)-O(2)	123.8(3)
C(13)-C(8)-C(9)	118.4(4)	O(1)-C(18)-C(17)	122.7(3)
C(13)-C(8)-C(7)	120.6(3)	O(2)-C(18)-C(17)	113.4(3)
C(9)-C(8)-C(7)	120.9(3)	O(3)-C(19)-O(4)	124.1(3)
C(10)-C(9)-C(8)	120.4(4)	O(3)-C(19)-N(1)	123.4(3)
C(10)-C(9)-H(9)	119.8	O(4)-C(19)-N(1)	112.6(3)
C(8)-C(9)-H(9)	119.8	O(4)-C(20)-C(22)	109.2(3)
C(11)-C(10)-C(9)	119.9(5)	O(4)-C(20)-C(23)	101.9(3)
C(11)-C(10)-H(10)	120.1	C(22)-C(20)-C(23)	111.2(4)
C(9)-C(10)-H(10)	120.1	O(4)-C(20)-C(21)	110.5(3)
C(10)-C(11)-C(12)	120.7(4)	C(22)-C(20)-C(21)	111.9(4)
C(10)-C(11)-H(11)	119.7	C(23)-C(20)-C(21)	111.6(4)
C(12)-C(11)-H(11)	119.7	C(20)-C(21)-H(21A)	109.5
C(11)-C(12)-C(13)	120.0(5)	C(20)-C(21)-H(21B)	109.5
C(11)-C(12)-H(12)	120.0	H(21A)-C(21)-H(21B)	109.5
C(13)-C(12)-H(12)	120.0	C(20)-C(21)-H(21C)	109.5
C(12)-C(13)-C(8)	120.6(4)	H(21A)-C(21)-H(21C)	109.5

H(21B)-C(21)-H(21C)	109.5	C(19)-N(1)-C(17)	122.9(3)
C(20)-C(22)-H(22A)	109.5	C(19)-N(1)-H(1A)	106.6
C(20)-C(22)-H(22B)	109.5	C(17)-N(1)-H(1A)	106.6
H(22A)-C(22)-H(22B)	109.5	C(18)-O(2)-H(2)	114(8)
C(20)-C(22)-H(22C)	109.5	C(19)-O(4)-C(20)	121.2(3)
H(22A)-C(22)-H(22C)	109.5	Cl(2)-C(24)-Cl(1)	114.7(4)
H(22B)-C(22)-H(22C)	109.5	Cl(2)-C(24)-Cl(3)	110.4(3)
C(20)-C(23)-H(23A)	109.5	Cl(1)-C(24)-Cl(3)	109.0(3)
C(20)-C(23)-H(23B)	109.5	Cl(2)-C(24)-H(24)	107.5
H(23A)-C(23)-H(23B)	109.5	Cl(1)-C(24)-H(24)	107.5
C(20)-C(23)-H(23C)	109.5	Cl(3)-C(24)-H(24)	107.5
H(23A)-C(23)-H(23C)	109.5		
H(23B)-C(23)-H(23C)	109.5		

cis-N,N-di-(tert-Butoxycarbonyl)-1-amino-1-methoxycarbonyl-2-(2,2-diphenylethenyl) cyclopropane 22.

A single crystal of **22** was coated in high-vacuum grease and mounted on a glass fibre. X-ray measurements were made using a Bruker SMART CCD area-detector diffractometer with Mo-K α radiation ($\lambda = 0.71073 \text{ \AA}$).¹ Intensities were integrated² from several series of exposures, each exposure covering 0.3° in θ , and the total data set being a sphere. Absorption corrections were applied, based on multiple and symmetry-equivalent measurements.³ The structure was solved by direct methods and refined by least squares on weighted F^2 values for all reflections.⁴ All non-hydrogen atoms were assigned anisotropic displacement parameters and refined without positional constraints. All hydrogen atoms were constrained to ideal geometries and refined with isotropic displacement parameters 1.5 times (methyl) or 1.2 times (all other hydrogen atoms) that of their parent atoms. Complex neutral-atom scattering factors were used.⁵

Table 3. Crystal data and structure refinement for **22**.

Identification code	LAA-22
Empirical formula	C ₂₉ H ₃₅ N O ₆
Formula weight	493.58
Temperature	173(2) K
Wavelength	0.71073 \AA
Crystal system	Triclinic
Space group	P1
Unit cell dimensions	a = 5.8851(13) \AA $\beta = 112.711(4)^\circ$ b = 10.029(2) \AA $\gamma = 91.376(4)^\circ$ c = 12.416(3) \AA $\alpha = 97.897(4)^\circ$
Volume	667.2(3) \AA^3
Z	1
Density (calculated)	1.228 Mg/m ³
Absorption coefficient	0.085 mm ⁻¹
F(000)	264
Crystal size	0.25 x 0.25 x 0.01 mm
θ range for data collection	2.23 to 24.13 $^\circ$
Index ranges	-6 $\leq h \leq$ 6, -11 $\leq k \leq$ 11, -14 $\leq l \leq$ 14
Reflections collected	5369
Independent reflections	4013 [$R_{\text{int}} = 0.0976$]
Completeness to $\theta = 24.13^\circ$	99.8 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	1 and 0.413
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	4013 / 9 / 332
Goodness-of-fit on F^2	S = 0.879

R indices [for 1800 reflections with $I > 2\sigma(I)$]	$R_1 = 0.0891$, $wR_2 = 0.1994$
R indices (for all 4013 data)	$R_1 = 0.1821$, $wR_2 = 0.2349$
Weighting scheme	$w^{-1} = \sigma^2(F_o^2) + (aP)^2$, where $P = [\max(F_o^2, 0) + 2F_c^2]/3$ $a = 0.1071$
Largest diff. peak and hole	0.394 and -0.406 eÅ ⁻³

Table 4. Bond lengths [Å] and angles [°] for **22**.

C(1)-C(2)	1.338(12)	C(16)-C(17)	1.547(12)
C(1)-C(6)	1.430(12)	C(16)-H(16A)	0.9900
C(1)-H(1)	0.9500	C(16)-H(16B)	0.9900
C(2)-C(3)	1.412(13)	C(17)-N	1.434(10)
C(2)-H(2)	0.9500	C(17)-C(18)	1.479(13)
C(3)-C(4)	1.406(13)	C(18)-O(1)	1.200(10)
C(3)-H(3)	0.9500	C(18)-O(2)	1.348(11)
C(4)-C(5)	1.339(13)	C(19)-O(2)	1.464(10)
C(4)-H(4)	0.9500	C(19)-H(19A)	0.9800
C(5)-C(6)	1.420(12)	C(19)-H(19B)	0.9800
C(5)-H(5)	0.9500	C(19)-H(19C)	0.9800
C(6)-C(7)	1.444(12)	C(20)-O(3)	1.230(11)
C(7)-C(14)	1.363(12)	C(20)-O(4)	1.296(10)
C(7)-C(8)	1.474(12)	C(20)-N	1.425(10)
C(8)-C(13)	1.376(12)	C(21)-C(24)	1.489(12)
C(8)-C(9)	1.429(13)	C(21)-C(23)	1.490(13)
C(9)-C(10)	1.387(13)	C(21)-O(4)	1.492(10)
C(9)-H(9)	0.9500	C(21)-C(22)	1.586(13)
C(10)-C(11)	1.345(14)	C(22)-H(22A)	0.9800
C(10)-H(10)	0.9500	C(22)-H(22B)	0.9800
C(11)-C(12)	1.401(15)	C(22)-H(22C)	0.9800
C(11)-H(11)	0.9500	C(23)-H(23A)	0.9800
C(12)-C(13)	1.411(13)	C(23)-H(23B)	0.9800
C(12)-H(12)	0.9500	C(23)-H(23C)	0.9800
C(13)-H(13)	0.9500	C(24)-H(24A)	0.9800
C(14)-C(15)	1.462(12)	C(24)-H(24B)	0.9800
C(14)-H(14)	0.9500	C(24)-H(24C)	0.9800
C(15)-C(16)	1.530(12)	C(25)-O(6)	1.234(10)
C(15)-C(17)	1.543(12)	C(25)-O(5)	1.276(10)
C(15)-H(15)	1.0000	C(25)-N	1.414(11)

C(26)-C(27)	1.482(13)	C(10)-C(9)-C(8)	119.2(10)
C(26)-C(29)	1.486(14)	C(10)-C(9)-H(9)	120.4
C(26)-O(5)	1.507(10)	C(8)-C(9)-H(9)	120.4
C(26)-C(28)	1.571(13)	C(11)-C(10)-C(9)	122.6(11)
C(27)-H(27A)	0.9800	C(11)-C(10)-H(10)	118.7
C(27)-H(27B)	0.9800	C(9)-C(10)-H(10)	118.7
C(27)-H(27C)	0.9800	C(10)-C(11)-C(12)	120.4(10)
C(28)-H(28A)	0.9800	C(10)-C(11)-H(11)	119.8
C(28)-H(28B)	0.9800	C(12)-C(11)-H(11)	119.8
C(28)-H(28C)	0.9800	C(11)-C(12)-C(13)	117.6(10)
C(29)-H(29A)	0.9800	C(11)-C(12)-H(12)	121.2
C(29)-H(29B)	0.9800	C(13)-C(12)-H(12)	121.2
C(29)-H(29C)	0.9800	C(8)-C(13)-C(12)	123.0(10)
		C(8)-C(13)-H(13)	118.5
C(2)-C(1)-C(6)	123.4(9)	C(12)-C(13)-H(13)	118.5
C(2)-C(1)-H(1)	118.3	C(7)-C(14)-C(15)	126.3(8)
C(6)-C(1)-H(1)	118.3	C(7)-C(14)-H(14)	116.8
C(1)-C(2)-C(3)	121.4(9)	C(15)-C(14)-H(14)	116.8
C(1)-C(2)-H(2)	119.3	C(14)-C(15)-C(16)	119.0(8)
C(3)-C(2)-H(2)	119.3	C(14)-C(15)-C(17)	120.0(7)
C(4)-C(3)-C(2)	115.5(9)	C(16)-C(15)-C(17)	60.4(5)
C(4)-C(3)-H(3)	122.3	C(14)-C(15)-H(15)	115.4
C(2)-C(3)-H(3)	122.3	C(16)-C(15)-H(15)	115.4
C(5)-C(4)-C(3)	123.6(10)	C(17)-C(15)-H(15)	115.4
C(5)-C(4)-H(4)	118.2	C(15)-C(16)-C(17)	60.2(6)
C(3)-C(4)-H(4)	118.2	C(15)-C(16)-H(16A)	117.8
C(4)-C(5)-C(6)	121.6(9)	C(17)-C(16)-H(16A)	117.8
C(4)-C(5)-H(5)	119.2	C(15)-C(16)-H(16B)	117.8
C(6)-C(5)-H(5)	119.2	C(17)-C(16)-H(16B)	117.8
C(5)-C(6)-C(1)	114.4(9)	H(16A)-C(16)-H(16B)	114.9
C(5)-C(6)-C(7)	123.0(9)	N-C(17)-C(18)	119.0(7)
C(1)-C(6)-C(7)	122.6(9)	N-C(17)-C(15)	117.0(7)
C(14)-C(7)-C(6)	120.3(8)	C(18)-C(17)-C(15)	116.8(7)
C(14)-C(7)-C(8)	122.5(9)	N-C(17)-C(16)	116.9(7)
C(6)-C(7)-C(8)	116.9(8)	C(18)-C(17)-C(16)	113.7(8)
C(13)-C(8)-C(9)	117.2(9)	C(15)-C(17)-C(16)	59.4(5)
C(13)-C(8)-C(7)	121.1(9)	O(1)-C(18)-O(2)	124.6(9)
C(9)-C(8)-C(7)	121.6(9)	O(1)-C(18)-C(17)	124.9(9)

O(2)-C(18)-C(17)	110.3(8)	O(6)-C(25)-O(5)	126.5(9)
O(2)-C(19)-H(19A)	109.5	O(6)-C(25)-N	118.5(7)
O(2)-C(19)-H(19B)	109.5	O(5)-C(25)-N	115.0(8)
H(19A)-C(19)-H(19B)	109.5	C(27)-C(26)-C(29)	115.6(8)
O(2)-C(19)-H(19C)	109.5	C(27)-C(26)-O(5)	102.1(8)
H(19A)-C(19)-H(19C)	109.5	C(29)-C(26)-O(5)	111.5(8)
H(19B)-C(19)-H(19C)	109.5	C(27)-C(26)-C(28)	109.0(9)
O(3)-C(20)-O(4)	126.8(8)	C(29)-C(26)-C(28)	110.6(8)
O(3)-C(20)-N	119.7(8)	O(5)-C(26)-C(28)	107.5(7)
O(4)-C(20)-N	113.4(8)	C(26)-C(27)-H(27A)	109.5
C(24)-C(21)-C(23)	114.6(9)	C(26)-C(27)-H(27B)	109.5
C(24)-C(21)-O(4)	104.3(7)	H(27A)-C(27)-H(27B)	109.5
C(23)-C(21)-O(4)	108.5(7)	C(26)-C(27)-H(27C)	109.5
C(24)-C(21)-C(22)	109.3(8)	H(27A)-C(27)-H(27C)	109.5
C(23)-C(21)-C(22)	110.4(7)	H(27B)-C(27)-H(27C)	109.5
O(4)-C(21)-C(22)	109.5(7)	C(26)-C(28)-H(28A)	109.5
C(21)-C(22)-H(22A)	109.5	C(26)-C(28)-H(28B)	109.5
C(21)-C(22)-H(22B)	109.5	H(28A)-C(28)-H(28B)	109.5
H(22A)-C(22)-H(22B)	109.5	C(26)-C(28)-H(28C)	109.5
C(21)-C(22)-H(22C)	109.5	H(28A)-C(28)-H(28C)	109.5
H(22A)-C(22)-H(22C)	109.5	H(28B)-C(28)-H(28C)	109.5
H(22B)-C(22)-H(22C)	109.5	C(26)-C(29)-H(29A)	109.5
C(21)-C(23)-H(23A)	109.5	C(26)-C(29)-H(29B)	109.5
C(21)-C(23)-H(23B)	109.5	H(29A)-C(29)-H(29B)	109.5
H(23A)-C(23)-H(23B)	109.5	C(26)-C(29)-H(29C)	109.5
C(21)-C(23)-H(23C)	109.5	H(29A)-C(29)-H(29C)	109.5
H(23A)-C(23)-H(23C)	109.5	H(29B)-C(29)-H(29C)	109.5
H(23B)-C(23)-H(23C)	109.5	C(25)-N-C(20)	125.8(7)
C(21)-C(24)-H(24A)	109.5	C(25)-N-C(17)	115.7(6)
C(21)-C(24)-H(24B)	109.5	C(20)-N-C(17)	115.0(7)
H(24A)-C(24)-H(24B)	109.5	C(18)-O(2)-C(19)	114.1(7)
C(21)-C(24)-H(24C)	109.5	C(20)-O(4)-C(21)	122.3(7)
H(24A)-C(24)-H(24C)	109.5	C(25)-O(5)-C(26)	121.4(8)
H(24B)-C(24)-H(24C)	109.5		

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