Anion Binding Behavior of Hybrid Calixpyrroles

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Supporting Information

I. ¹H NMR titration studies	S1
II. ITC titration studies.....	S7
III. X-ray experimental section.....	S8

I. ¹H NMR titration studies

Proton NMR-based titration studies were carried out using a Varian Unity Plus 400 MHz NMR spectrometer. The receptor solutions were titrated by adding known quantities of a concentrated solution of the anions in question. All anions were used in the form of their tetrabutylammonium salts. The anion solutions used to effect the titration contained the receptors at the same concentration as the receptor solutions into which they were being titrated so as to obviate a need to account for dilution effects during the titrations. The data were fit to a

1:1 binding profile according to the method of Wilcox¹ using the changes in the pyrrolic NH resonances in the ¹H NMR spectra.

¹H NMR titrations for compound 2 (Figure 1-3)

¹H NMR titrations for compound 3 (Figure 4-5)

¹H NMR titrations for compound 4 (Figure 6-7)

¹H NMR titrations for compound 5 (Figure 8-9)

Fig. 8 ¹H NMR spectroscopic titration curve for compound **5** and fluoride anion in CD₃CN. **[5]** = 2×10^{-3} M. **[F]** = 0 – 9.45×10^{-3} M. The curve shows the fit of the experimental data to a 1:1 binding profile.

Fig. 9 ¹H NMR spectroscopic titration curve for compound **5** and benzoate anion in CD₃CN. **[5]** = 2.72×10^{-3} M. **[PhCO₂]⁻** = 0 – 42.6×10^{-3} M. The curve shows the fit of the experimental data to a 1:1 binding profile.

¹H NMR titrations for compound 6 and 7 (Figure 10-11)

Fig. 10 ¹H NMR spectroscopic titration curve for compound **6** and dihydrogen phosphate anion in CD₃CN. **[6]** = 2×10^{-3} M. **[H₂PO₄]** = 0 – 40.3×10^{-3} M. The curve shows the fit of the experimental data to a 1:1 binding profile.

Fig. 11 ¹H NMR spectroscopic titration curve for compound **7** and dihydrogen phosphate anion in CD₃CN. **[7]** = 2×10^{-3} M. **[H₂PO₄]** = 0 – 38.4×10^{-3} M. The curve shows the fit of the experimental data to a 1:1 binding profile.

¹H NMR titrations for compound 8 (Figure 12-14)

References:

1. Wilcox, C. S. in *Frontiers in Supramolecular Organic Chemistry and Photochemistry*; Schneider, H. J., Dürr, H., Eds.; VCH: Weinheim, **1991**.

II. ITC titration studies

Microcalorimetric titrations (experimental temperature: 30 °C) were performed using an isothermal titration calorimeter (ITC) purchased from Microcal Inc., MA. The ORIGIN software provided by Microcal Inc. was used to calculate the binding constant (K_a) and the enthalpy change (ΔH). The solvent, CH_3CN , was HPLC grade (Fisher) but was not further dried or purified before use.

III. X-ray experimental section

Crystallographic Material for $\mathbf{8 \cdot 2CH_2Cl_2}$:

- X-ray Experimental.
- Table 1. Crystallographic Data for $\mathbf{8 \cdot 2CH_2Cl_2}$.
- Table 2. Fractional coordinates and equivalent isotropic thermal parameters ($\text{\AA}^2$) for the non-hydrogen atoms of $\mathbf{8 \cdot 2CH_2Cl_2}$.
- Table 3. Bond Lengths ($\text{\AA}$) and Angles ($^\circ$) for the non-hydrogen atoms of $\mathbf{8 \cdot 2CH_2Cl_2}$.
- Table 4. Anisotropic thermal parameters for the non-hydrogen atoms of $\mathbf{8 \cdot 2CH_2Cl_2}$.
- Table 5. Fractional coordinates and isotropic thermal parameters ($\text{\AA}^2$) for the hydrogen atoms of $\mathbf{8 \cdot 2CH_2Cl_2}$.
- Table 6. Torsion Angles ($^\circ$) for the non-hydrogen atoms of $\mathbf{8 \cdot 2CH_2Cl_2}$.
- Figure 1. View of the macrocycle in $\mathbf{8 \cdot 2CH_2Cl_2}$ showing the atom labeling scheme.
- Figure 2. View illustrating the close interactions between the methylene chloride solvent molecules and the macrocycle in $\mathbf{8 \cdot 2CH_2Cl_2}$.
- Figure 3. Unit cell packing diagram for $\mathbf{8 \cdot 2CH_2Cl_2}$.

X-ray Experimental for **8**·2CH₂Cl₂: C₃₈H₄₄Cl₄N₄OS, crystals grew as colorless prisms by vapor diffusion of hexanes into a dichloromethane solution of the macrocycle. The data crystal was cut from a larger crystal and had approximate dimensions; 0.24 × 0.22 × 0.20 mm. The data were collected on a Nonius Kappa CCD diffractometer using a graphite monochromator with MoK α radiation (λ = 0.71073 Å). A total of 418 frames of data were collected using ω -scans with a scan range of 1° and a counting time of 41 seconds per frame. The data were collected at 153 K using an Oxford Cryostream low temperature device. Details of crystal data, data collection and structure refinement are listed in Table 1. Data reduction were performed using DENZO-SMN.¹ The structure was solved by direct methods using SIR97² and refined by full-matrix least-squares on F² with anisotropic displacement parameters for the non-H atoms using SHELXL-97.³ The hydrogen atoms on carbon were calculated in ideal positions with isotropic displacement parameters set to 1.2 × U_{eq} of the attached atom (1.5 × U_{eq} for methyl hydrogen atoms). The hydrogen atoms on the pyrrole nitrogen atoms and on the solvent molecule were observed in a ΔF map and refined with isotropic displacement parameters.

The macrocycle resides around a crystallographic inversion center at $\frac{1}{2}, \frac{1}{2}, \frac{1}{2}$. The presence of the inversion center requires that the thiophene ring and the furan ring be disordered. In the present crystal, it appeared that only the positions of the sulfur and oxygen atoms were affected by the disorder. During the course of the refinement, the sulfur and oxygen atoms were refined without restraints with a site occupancy factor fixed at $\frac{1}{2}$.

The function, $\Sigma w(|F_o|^2 - |F_c|^2)^2$, was minimized, where $w = 1/[(\sigma(F_o))^2 + (0.0483 \cdot P)^2 + (0.2576 \cdot P)]$ and $P = (|F_o|^2 + 2|F_c|^2)/3$. $R_w(F^2)$ refined to 0.117, with $R(F)$ equal to 0.0467 and a goodness of fit, S , = 1.04. Definitions used for calculating $R(F)$, $R_w(F^2)$ and the goodness of fit, S , are given below.⁴ The data were corrected for secondary extinction effects. The correction

takes the form: $F_{\text{corr}} = kF_c/[1 + (9.0(11) \times 10^{-5}) * F_c^2 \lambda^3/(\sin 2\theta)]^{0.25}$ where k is the overall scale factor. Neutral atom scattering factors and values used to calculate the linear absorption coefficient are from the International Tables for X-ray Crystallography (1992).⁵ All figures were generated using SHELXTL/PC.⁶ Tables of positional and thermal parameters, bond lengths and angles, torsion angles, figures and lists of observed and calculated structure factors are located in tables 1 through 7.

References

1. DENZO-SMN. (1997). Z. Otwinowski and W. Minor, Methods in Enzymology, **276**: Macromolecular Crystallography, part A, 307 – 326, C. W. Carter, Jr. and R. M. Sweets, Editors, Academic Press.
2. SIR97. (1999). A program for crystal structure solution. Altomare A., Burla M.C., Camalli M., Cascarano G.L., Giacovazzo C. , Guagliardi A., Moliterni A.G.G., Polidori G., Spagna R. J. Appl. Cryst. 32, 115-119.
3. Sheldrick, G. M. (1994). SHELXL97. Program for the Refinement of Crystal Structures. University of Gottingen, Germany.
4. $R_w(F^2) = \{ \sum w(|F_o|^2 - |F_c|^2)^2 / \sum w(|F_o|^4) \}^{1/2}$ where w is the weight given each reflection.
 $R(F) = \sum (|F_o| - |F_c|) / \sum |F_o|$ for reflections with $F_o > 4(\sigma(F_o))$.
 $S = [\sum w(|F_o|^2 - |F_c|^2)^2 / (n - p)]^{1/2}$, where n is the number of reflections and p is the number of refined parameters.
5. International Tables for X-ray Crystallography (1992). Vol. C, Tables 4.2.6.8 and 6.1.1.4, A. J. C. Wilson, editor, Boston: Kluwer Academic Press.
6. Sheldrick, G. M. (1994). SHELXTL/PC (Version 5.03). Siemens Analytical X-ray Instruments, Inc., Madison, Wisconsin, USA.

Table 1. Crystal data and structure refinement for **8·2CH₂Cl₂**.

Empirical formula	C ₃₈ H ₄₄ Cl ₄ N ₄ O S	
Formula weight	746.63	
Temperature	153(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	<i>P</i> 2 ₁ /n	
Unit cell dimensions	<i>a</i> = 10.4748(2) Å	α = 90°.
	<i>b</i> = 13.6047(2) Å	β = 92.435(1)°.
	<i>c</i> = 13.2513(2) Å	γ = 90°.
Volume	1886.69(5) Å ³	
<i>Z</i>	2	
Density (calculated)	1.314 g m ⁻³	
Absorption coefficient	0.405 mm ⁻¹	
<i>F</i> (000)	784	
Crystal size	0.24 × 0.22 × 0.20 mm	
Theta range for data collection	2.94 to 27.48°.	
Index ranges	-13 ≤ <i>h</i> ≤ 13, -16 ≤ <i>k</i> ≤ 17, -17 ≤ <i>l</i> ≤ 17	
Reflections collected	7911	
Independent reflections	4314 [<i>R</i> (int) = 0.0414]	
Completeness to θ = 27.48°	99.5 %	
Absorption correction	None	
Refinement method	Full-matrix least-squares on <i>F</i> ²	
Data / restraints / parameters	4314 / 0 / 243	
Goodness-of-fit on <i>F</i> ²	1.044	
Final <i>R</i> indices [<i>I</i> > 2σ(<i>I</i>)]	<i>R</i> 1 = 0.0467, <i>wR</i> 2 = 0.1002	
<i>R</i> indices (all data)	<i>R</i> 1 = 0.1085, <i>wR</i> 2 = 0.1170	
Extinction coefficient	9.0(11) × 10 ⁻⁶	
Largest diff. peak and hole	0.287 and -0.274 e.Å ⁻³	

Table 2. Atomic coordinates ($\times 10^4$) and equivalent isotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for **8**·2CH₂Cl₂. U(eq) is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U(eq)
Cl1A	5418(1)	4151(1)	6310(1)	43(1)
Cl2A	5072(1)	5609(1)	7893(1)	60(1)
C1A	5039(3)	5390(2)	6587(2)	39(1)
S1	5780(2)	2160(2)	3765(1)	34(1)
O1	5852(6)	2144(5)	4215(3)	35(1)
N1	8094(2)	4231(1)	4908(2)	29(1)
N2	7243(2)	6825(1)	5006(2)	31(1)
C1	4852(2)	1714(2)	4722(2)	35(1)
C2	5452(2)	1537(2)	5621(2)	39(1)
C3	6774(2)	1800(2)	5597(2)	39(1)
C4	7039(2)	2154(2)	4685(2)	34(1)
C5	8273(2)	2508(2)	4253(2)	36(1)
C6	8237(2)	3612(2)	4107(2)	31(1)
C7	8351(2)	4192(2)	3275(2)	39(1)
C8	8277(2)	5188(2)	3582(2)	37(1)
C9	8102(2)	5197(2)	4597(2)	29(1)
C10	7912(2)	5993(2)	5304(2)	29(1)
C11	8287(2)	6124(2)	6295(2)	36(1)
C12	7828(2)	7051(2)	6604(2)	38(1)
C13	7186(2)	7470(2)	5801(2)	34(1)
C14	6503(2)	8451(2)	5662(2)	40(1)
C15	8466(3)	2016(2)	3221(2)	70(1)
C16	9382(2)	2230(2)	4976(2)	46(1)
C17	6517(3)	8986(2)	6683(2)	75(1)
C18	7204(2)	9080(2)	4905(2)	48(1)

Table 3. Bond lengths [Å] and angles [°] for **8**·2CH₂Cl₂.

Cl1A-C1A	1.773(3)	C7-H7	0.96
Cl2A-C1A	1.755(2)	C8-C9	1.366(3)
C1A-H1AA	0.95(3)	C8-H8	0.96
C1A-H1AB	0.98(2)	C9-C10	1.451(3)
S1-C1	1.740(3)	C10-C11	1.366(3)
S1-C4	1.758(3)	C11-C12	1.417(3)
O1-C4	1.367(6)	C11-H11	0.96
O1-C1	1.397(6)	C12-C13	1.360(3)
N1-C6	1.367(3)	C12-H12	0.96
N1-C9	1.376(3)	C13-C14	1.521(3)
N1-H1N	0.86(2)	C14-C1#1	1.504(3)
N2-C13	1.376(3)	C14-C18	1.530(3)
N2-C10	1.380(3)	C14-C17	1.536(3)
N2-H2N	0.84(2)	C15-H15A	0.96
C1-C2	1.345(3)	C15-H15B	0.96
C1-C14#1	1.504(3)	C15-H15C	0.96
C2-C3	1.431(3)	C16-H16A	0.96
C2-H2	0.96	C16-H16B	0.96
C3-C4	1.341(3)	C16-H16C	0.96
C3-H3	0.96	C17-H17A	0.96
C4-C5	1.514(3)	C17-H17B	0.96
C5-C6	1.516(3)	C17-H17C	0.96
C5-C16	1.521(3)	C18-H18A	0.96
C5-C15	1.543(3)	C18-H18B	0.96
C6-C7	1.365(3)	C18-H18C	0.96
C7-C8	1.418(3)		
Cl2A-C1A-Cl1A	111.79(14)	C4-O1-C1	118.1(3)
Cl2A-C1A-H1AA	109.0(14)	C6-N1-C9	110.70(18)
Cl1A-C1A-H1AA	106.4(14)	C6-N1-H1N	127.9(17)
Cl2A-C1A-H1AB	109.4(14)	C9-N1-H1N	120.4(17)
Cl1A-C1A-H1AB	107.6(14)	C13-N2-C10	110.0(2)
H1AA-C1A-H1AB	113(2)	C13-N2-H2N	126.7(17)
C1-S1-C4	85.32(14)	C10-N2-H2N	122.7(17)

C2-C1-O1	99.8(3)	N2-C10-C9	120.6(2)
C2-C1-C14#1	132.2(2)	C10-C11-C12	107.9(2)
O1-C1-C14#1	128.0(3)	C10-C11-H11	125.7
C2-C1-S1	116.98(19)	C12-C11-H11	126.4
C14#1-C1-S1	110.70(18)	C13-C12-C11	108.1(2)
C1-C2-C3	110.6(2)	C13-C12-H12	125.5
C1-C2-H2	124.1	C11-C12-H12	126.5
C3-C2-H2	125.4	C12-C13-N2	107.2(2)
C4-C3-C2	110.4(2)	C12-C13-C14	132.6(2)
C4-C3-H3	124.3	N2-C13-C14	120.2(2)
C2-C3-H3	125.3	C1#1-C14-C13	109.98(18)
C3-C4-O1	100.8(3)	C1#1-C14-C18	109.50(19)
C3-C4-C5	131.9(2)	C13-C14-C18	109.50(18)
O1-C4-C5	127.3(3)	C1#1-C14-C17	110.0(2)
C3-C4-S1	116.65(19)	C13-C14-C17	108.8(2)
C5-C4-S1	111.32(18)	C18-C14-C17	109.0(2)
C4-C5-C6	110.25(17)	C5-C15-H15A	108.8
C4-C5-C16	108.91(19)	C5-C15-H15B	110.7
C6-C5-C16	109.92(19)	H15A-C15-H15B	109.5
C4-C5-C15	110.1(2)	C5-C15-H15C	108.9
C6-C5-C15	108.70(19)	H15A-C15-H15C	109.5
C16-C5-C15	109.0(2)	H15B-C15-H15C	109.5
C7-C6-N1	106.70(19)	C5-C16-H16A	109.0
C7-C6-C5	132.3(2)	C5-C16-H16B	109.0
N1-C6-C5	120.99(19)	H16A-C16-H16B	109.5
C6-C7-C8	108.2(2)	C5-C16-H16C	110.4
C6-C7-H7	125.3	H16A-C16-H16C	109.5
C8-C7-H7	126.5	H16B-C16-H16C	109.5
C9-C8-C7	107.6(2)	C14-C17-H17A	108.6
C9-C8-H8	125.8	C14-C17-H17B	109.0
C7-C8-H8	126.6	H17A-C17-H17B	109.5
C8-C9-N1	106.83(19)	C14-C17-H17C	110.9
C8-C9-C10	132.0(2)	H17A-C17-H17C	109.5
N1-C9-C10	121.10(19)	H17B-C17-H17C	109.5
C11-C10-N2	106.87(19)	C14-C18-H18A	109.4
C11-C10-C9	132.5(2)	C14-C18-H18B	110.0

H18A-C18-H18B	109.5	H18A-C18-H18C	109.5
C14-C18-H18C	109.0	H18B-C18-H18C	109.5

Symmetry transformations used to generate equivalent atoms:

#1 -x+1,-y+1,-z+1

Table 4. Anisotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for **8**·2CH₂Cl₂. The anisotropic displacement factor exponent takes the form: $-2\pi^2 [h^2 a^{*2} U^{11} + \dots + 2 h k a^* b^* U^{12}]$.

	U ¹¹	U ²²	U ³³	U ²³	U ¹³	U ¹²
Cl1A	37(1)	42(1)	52(1)	-7(1)	6(1)	-3(1)
Cl2A	74(1)	62(1)	44(1)	-13(1)	2(1)	-5(1)
C1A	34(1)	40(2)	44(2)	-4(1)	2(1)	-3(1)
S1	28(1)	40(1)	33(1)	5(1)	1(1)	-2(1)
O1	39(2)	35(2)	30(3)	2(3)	2(3)	-1(2)
N1	29(1)	27(1)	32(1)	1(1)	3(1)	1(1)
N2	33(1)	27(1)	34(1)	-1(1)	-5(1)	2(1)
C1	37(1)	24(1)	45(1)	4(1)	14(1)	4(1)
C2	34(1)	34(1)	51(2)	2(1)	8(1)	4(1)
C3	34(1)	35(1)	48(2)	-1(1)	0(1)	3(1)
C4	30(1)	23(1)	50(2)	-4(1)	-8(1)	5(1)
C5	42(1)	28(1)	38(1)	-2(1)	6(1)	5(1)
C6	29(1)	31(1)	34(1)	-3(1)	3(1)	4(1)
C7	38(1)	43(2)	36(1)	2(1)	8(1)	8(1)
C8	35(1)	34(1)	43(2)	8(1)	7(1)	6(1)
C9	20(1)	24(1)	43(1)	3(1)	0(1)	0(1)
C10	22(1)	24(1)	42(1)	3(1)	-1(1)	-3(1)
C11	33(1)	30(1)	43(1)	8(1)	-7(1)	-2(1)
C12	44(1)	37(1)	34(1)	0(1)	-4(1)	-2(1)
C13	37(1)	28(1)	36(1)	-4(1)	1(1)	-2(1)
C14	47(2)	29(1)	43(1)	-4(1)	-2(1)	5(1)
C15	116(3)	40(2)	54(2)	-14(1)	22(2)	7(2)
C16	32(1)	34(1)	73(2)	10(1)	5(1)	8(1)
C17	122(3)	49(2)	53(2)	-20(2)	-20(2)	35(2)
C18	37(1)	32(1)	74(2)	4(1)	-2(1)	-5(1)

Table 5. Hydrogen coordinates ($\times 10^4$) and isotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for **8**·2CH₂Cl₂.

	x	y	z	U(eq)
H2	5040	1275	6196	47
H3	7389	1733	6150	47
H7	8461	3958	2601	47
H8	8344	5757	3159	45
H11	8775	5666	6705	43
H12	7939	7336	7265	46
H15A	7772	2200	2764	104
H15B	9260	2220	2951	104
H15C	8470	1315	3307	104
H16A	9270	2545	5614	69
H16B	9387	1530	5067	69
H16C	10177	2435	4710	69
H17A	6068	8591	7152	113
H17B	7386	9070	6926	113
H17C	6112	9617	6617	113
H18A	7184	8755	4262	71
H18B	6800	9711	4836	71
H18C	8075	9164	5145	71
H1AA	4190(20)	5500(17)	6315(18)	46(7)
H1AB	5680(20)	5809(18)	6273(18)	53(7)
H1N	7880(20)	4082(18)	5512(19)	49(8)
H2N	6850(20)	6872(18)	4443(18)	44(8)

Table 6. Torsion angles [°] for **8**·2CH₂Cl₂.

C4-O1-C1-C2	6.5(5)	C5-C6-C7-C8	-178.5(2)
C4-O1-C1-C14#1	-175.2(3)	C6-C7-C8-C9	-0.8(3)
C4-S1-C1-C2	-2.9(2)	C7-C8-C9-N1	1.1(2)
C4-S1-C1-O1	15.9(10)	C7-C8-C9-C10	-177.2(2)
C4-S1-C1-C14#1	-179.73(17)	C6-N1-C9-C8	-1.1(2)
O1-C1-C2-C3	-3.7(3)	C6-N1-C9-C10	177.43(18)
C14#1-C1-C2-C3	178.1(2)	C13-N2-C10-C11	-0.5(2)
S1-C1-C2-C3	2.1(3)	C13-N2-C10-C9	179.84(18)
C1-C2-C3-C4	0.4(3)	C8-C9-C10-C11	-145.4(3)
C2-C3-C4-O1	3.3(3)	N1-C9-C10-C11	36.5(3)
C2-C3-C4-C5	-178.2(2)	C8-C9-C10-N2	34.2(3)
C2-C3-C4-S1	-2.6(3)	N1-C9-C10-N2	-143.9(2)
C1-O1-C4-C3	-6.3(5)	N2-C10-C11-C12	0.4(2)
C1-O1-C4-C5	175.1(3)	C9-C10-C11-C12	180.0(2)
C1-S1-C4-C3	3.1(2)	C10-C11-C12-C13	-0.1(3)
C1-S1-C4-C5	179.60(17)	C11-C12-C13-N2	-0.2(3)
C3-C4-C5-C6	-109.7(3)	C11-C12-C13-C14	-179.2(2)
O1-C4-C5-C6	68.4(4)	C10-N2-C13-C12	0.4(2)
S1-C4-C5-C6	74.6(2)	C10-N2-C13-C14	179.59(19)
C3-C4-C5-C16	11.0(3)	C12-C13-C14-C1#1	-125.2(3)
O1-C4-C5-C16	-170.9(4)	N2-C13-C14-C1#1	55.9(3)
S1-C4-C5-C16	-164.76(18)	C12-C13-C14-C18	114.4(3)
C3-C4-C5-C15	130.4(3)	N2-C13-C14-C18	-64.5(3)
O1-C4-C5-C15	-51.5(4)	C12-C13-C14-C17	-4.6(4)
S1-C4-C5-C15	-45.4(2)	N2-C13-C14-C17	176.5(2)
C9-N1-C6-C7	0.6(2)		
C9-N1-C6-C5	179.37(18)		
C4-C5-C6-C7	-121.4(3)	Symmetry transformations used to generate equivalent atoms: #1 -x+1,-y+1,-z+1	
C16-C5-C6-C7	118.6(3)		
C15-C5-C6-C7	-0.6(4)		
C4-C5-C6-N1	60.2(3)		
C16-C5-C6-N1	-59.8(3)		
C15-C5-C6-N1	-179.0(2)		
N1-C6-C7-C8	0.1(2)		

Figure 1. View of the macrocycle in **8**·2CH₂Cl₂ showing the atom labeling scheme. Displacement ellipsoids are scaled to the 50% probability level. The methyl hydrogen atoms have been removed for clarity. The macrocycle lies around a crystallographic inversion center at $\frac{1}{2}, \frac{1}{2}, \frac{1}{2}$. Atoms with labels appended by a ' are related by $1 - x, 1 - y, 1 - z$. The positions of the oxygen atom and the sulfur atom are disordered by the center of symmetry.

Figure 2. View illustrating the close interactions between the methylene chloride solvent molecules and the macrocycle in $8 \cdot 2\text{CH}_2\text{Cl}_2$. Displacement ellipsoids are scaled to the 30% probability level. Most hydrogen atoms have been removed for clarity. The macrocycle lies around a crystallographic inversion center at $\frac{1}{2}, \frac{1}{2}, \frac{1}{2}$. The positions of the oxygen atom and the sulfur atom are disordered by the center of symmetry. Dashed lines are indicative of close contacts. The geometry of these interactions are: N1-H1N $\cdots$ Cl1A, N $\cdots$ Cl 3.430(2)Å, H $\cdots$ Cl 2.83(2)Å, N-H $\cdots$ Cl 128(2)°; N2-H2N $\cdots$ Cl1A (related by 1-x, 1-y, 1-z), N $\cdots$ Cl 3.486(2)Å, H $\cdots$ Cl 2.90(2)Å, N-H $\cdots$ Cl 129(2)°. The hydrogen atoms on the solvent molecule are in close contact with the ring center of the pyrrole rings; H1AA $\cdots$ ring center (N1, C6, C7, C8 and C9 related by 1-x, 1-y, 1-z) 2.55(3)Å, H1AB $\cdots$ ring center (N2, C10, C11, C12, C13), 2.53(2)Å.

Figure 3 Unit cell packing diagram for **8**·2CH₂Cl₂. The view is approximately down the **c** axis.

Crystallographic Material for $7 \cdot (\text{C}_4\text{H}_9)_4\text{NPhCO}_2 \cdot \text{CH}_2\text{Cl}_2$

- X-ray Experimental.
- Table 1. Crystallographic Data for $7 \cdot (\text{C}_4\text{H}_9)_4\text{N PhCO}_2 \cdot \text{CH}_2\text{Cl}_2$.
- Table 2. Fractional coordinates and equivalent isotropic thermal parameters ($\text{\AA}^2$) for the non-hydrogen atoms of $7 \cdot (\text{C}_4\text{H}_9)_4\text{N PhCO}_2 \cdot \text{CH}_2\text{Cl}_2$.
- Table 3. Bond Lengths ($\text{\AA}$) and Angles ($^\circ$) for the non-hydrogen atoms of $7 \cdot (\text{C}_4\text{H}_9)_4\text{N PhCO}_2 \cdot \text{CH}_2\text{Cl}_2$.
- Table 4. Anisotropic thermal parameters for the non-hydrogen atoms of $7 \cdot (\text{C}_4\text{H}_9)_4\text{N PhCO}_2 \cdot \text{CH}_2\text{Cl}_2$.
- Table 5. Fractional coordinates and isotropic thermal parameters ($\text{\AA}^2$) for the hydrogen atoms of $7 \cdot (\text{C}_4\text{H}_9)_4\text{N PhCO}_2 \cdot \text{CH}_2\text{Cl}_2$.
- Table 6. Torsion Angles ($^\circ$) for the non-hydrogen atoms of $7 \cdot (\text{C}_4\text{H}_9)_4\text{N PhCO}_2 \cdot \text{CH}_2\text{Cl}_2$.
- Table 7. Hydrogen Bond Lengths ($\text{\AA}$) and Angles ($^\circ$) for $7 \cdot (\text{C}_4\text{H}_9)_4\text{N PhCO}_2 \cdot \text{CH}_2\text{Cl}_2$.
- Figure 1. View of the macrocycle in $7 \cdot (\text{C}_4\text{H}_9)_4\text{N PhCO}_2 \cdot \text{CH}_2\text{Cl}_2$ showing the atom labeling scheme.
- Figure 2. View illustrating the H-bonding interaction between the benzoate anion and the macrocycle in $7 \cdot (\text{C}_4\text{H}_9)_4\text{N PhCO}_2 \cdot \text{CH}_2\text{Cl}_2$.
- Figure 3. View of the cation in $7 \cdot (\text{C}_4\text{H}_9)_4\text{N PhCO}_2 \cdot \text{CH}_2\text{Cl}_2$ showing the atom labeling scheme.
- Figure 4. Unit cell packing diagram for $7 \cdot (\text{C}_4\text{H}_9)_4\text{N PhCO}_2 \cdot \text{CH}_2\text{Cl}_2$.

X-ray Experimental for **7**·(C₄H₉)₄N PhCO₂·CH₂Cl₂: C₆₀H₈₃Cl₂N₅O₂S₂, crystals grew as clusters of colorless lathes by slow evaporation of a dichloromethane solution containing **7** and tetrabutylammonium benzoate (1:1 molar ratio). The data crystal was a long lathe that had approximate dimensions; 0.55 × 0.18 × 0.10 mm. The data were collected on a Nonius Kappa CCD diffractometer using a graphite monochromator with MoK α radiation (λ = 0.71073Å). A total of 338 frames of data were collected using ω -scans with a scan range of 1° and a counting time of 204 seconds per frame. The data were collected at 153 K using an Oxford Cryostream low temperature device. Details of crystal data, data collection and structure refinement are listed in Table 1. Data reduction were performed using DENZO-SMN.¹ The structure was solved by direct methods using SIR97² and refined by full-matrix least-squares on F² with anisotropic displacement parameters for the non-H atoms using SHELXL-97.³ The hydrogen atoms on carbon were calculated in ideal positions with isotropic displacement parameters set to 1.2 × U_{eq} of the attached atom (1.5 × U_{eq} for methyl hydrogen atoms). The hydrogen atoms on the pyrrolic nitrogen atoms were observed in a ΔF map and refined with isotropic displacement parameters. One of the methyl carbon atoms of the tetrakis-n-butyl ammonium ion was found to be disordered about two orientations. The site occupancy factor for the two orientations given by atoms C4b and C4ba was refined with variable x set to the site occupancy factor for C4b and (1-x) set to the site occupancy factor for C4ba. While the site occupancy factors were being refined, a common isotropic displacement parameter was refined for both atoms. In this way, the site occupancy factor for C4b refined to 58(2)%. The geometry around the disordered methyl carbons was restrained to be approximately equivalent throughout the refinement procedure.

The direction of the two-fold screw rotation axis was determined by the method of Flack.⁴ The Flack x parameter refined to 0.0(6) for the reported configuration. The Flack parameter for the inverted structure refined to 1.04(6).

The function, $\sum w(|F_o|^2 - |F_c|^2)^2$, was minimized, where $w = 1/[(\sigma(F_o))^2 + (0.0279 \cdot P)^2 + (1.70 \cdot P)]$ and $P = (|F_o|^2 + 2|F_c|^2)/3$. $R_w(F^2)$ refined to 0.107, with $R(F)$ equal to 0.0507 and a goodness of fit, S , = 0.99. Definitions used for calculating $R(F)$, $R_w(F^2)$ and the goodness of fit, S , are given below.⁵ The data were checked for secondary extinction affects but no correction was necessary. Neutral atom scattering factors and values used to calculate the linear absorption coefficient are from the International Tables for X-ray Crystallography (1992).⁶ All figures were generated using SHELXTL/PC.⁷ Tables of positional and thermal parameters, bond lengths and angles, torsion angles, H-bonding interactions, figures and lists of observed and calculated structure factors are located in tables 1 through 7.

References

1. DENZO-SMN. (1997). Z. Otwinowski and W. Minor, Methods in Enzymology, **276**: Macromolecular Crystallography, part A, 307 – 326, C. W. Carter, Jr. and R. M. Sweets, Editors, Academic Press.
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4. Flack, H. D. (1983). Acta Cryst. A39, 876-881.

5. $R_w(F^2) = \{ \Sigma w(|F_o|^2 - |F_c|^2)^2 / \Sigma w(|F_o|)^4 \}^{1/2}$ where w is the weight given each reflection.

$$R(F) = \Sigma (|F_o| - |F_c|) / \Sigma |F_o| \text{ for reflections with } F_o > 4(\sigma(F_o)).$$

$$S = [\Sigma w(|F_o|^2 - |F_c|^2)^2 / (n - p)]^{1/2}, \text{ where } n \text{ is the number of reflections and } p \text{ is the number of refined parameters.}$$

6. International Tables for X-ray Crystallography (1992). Vol. C, Tables 4.2.6.8 and 6.1.1.4, A. J. C. Wilson, editor, Boston: Kluwer Academic Press.
7. Sheldrick, G. M. (1994). SHELXTL/PC (Version 5.03). Siemens Analytical X-ray Instruments, Inc., Madison, Wisconsin, USA.

Table 1. Crystal data and structure refinement for **7**·(C₄H₉)₄NPhCO₂·CH₂Cl₂.

Empirical formula	C ₆₀ H ₈₃ Cl ₂ N ₅ O ₂ S ₂	
Formula weight	1041.33	
Temperature	153(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	<i>P</i> 2 ₁	
Unit cell dimensions	<i>a</i> = 11.5806(2) Å	α = 90°.
	<i>b</i> = 22.8486(4) Å	β = 113.6230(10)°.
	<i>c</i> = 12.1009(2) Å	γ = 90°.
Volume	2933.59(9) Å ³	
<i>Z</i>	2	
Density (calculated)	1.179 g cm ⁻³	
Absorption coefficient	0.227 mm ⁻¹	
<i>F</i> (000)	1120	
Crystal size	0.55 × 0.18 × 0.10 mm	
Theta range for data collection	3.14 to 27.49°.	
Index ranges	-15 ≤ <i>h</i> ≤ 15, -20 ≤ <i>k</i> ≤ 29, -15 ≤ <i>l</i> ≤ 15	
Reflections collected	10518	
Independent reflections	10518	
Completeness to theta = 27.49°	99.3 %	
Absorption correction	None	
Refinement method	Full-matrix least-squares on <i>F</i> ²	
Data / restraints / parameters	10518 / 3 / 668	
Goodness-of-fit on <i>F</i> ²	0.986	
Final <i>R</i> indices [<i>I</i> > 2σ(<i>I</i>)]	<i>R</i> 1 = 0.0507, <i>wR</i> 2 = 0.0909	
<i>R</i> indices (all data)	<i>R</i> 1 = 0.0988, <i>wR</i> 2 = 0.1074	
Absolute structure parameter	0.00(6)	
Largest diff. peak and hole	0.276 and -0.297 e.Å ⁻³	

Table 2. Atomic coordinates ($\times 10^4$) and equivalent isotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for $7 \cdot (\text{C}_4\text{H}_9)_4\text{NPhCO}_2 \cdot \text{CH}_2\text{Cl}_2$. U(eq) is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U(eq)
S1	8495(1)	4286(1)	7238(1)	39(1)
S2	1551(1)	4042(1)	2397(1)	38(1)
N1	6111(3)	2777(1)	6811(2)	29(1)
N2	3557(3)	2681(1)	4956(2)	28(1)
N3	4780(3)	4697(1)	1722(3)	30(1)
N4	7345(3)	4738(1)	3466(2)	29(1)
C1	8846(3)	4269(2)	5984(3)	29(1)
C2	8816(3)	3711(2)	5602(3)	40(1)
C3	8547(3)	3297(2)	6336(3)	42(1)
C4	8366(3)	3535(2)	7278(3)	34(1)
C5	8113(3)	3220(2)	8272(3)	39(1)
C6	7273(3)	2707(2)	7743(3)	31(1)
C7	7391(3)	2121(2)	8020(3)	39(1)
C8	6279(3)	1835(2)	7235(3)	34(1)
C9	5501(3)	2251(1)	6499(3)	26(1)
C10	4221(3)	2203(1)	5581(3)	25(1)
C11	3429(3)	1729(2)	5206(3)	35(1)
C12	2267(3)	1923(2)	4329(3)	37(1)
C13	2367(3)	2515(1)	4187(3)	30(1)
C14	1401(3)	2963(2)	3468(3)	34(1)
C15	1881(3)	3307(1)	2659(3)	29(1)
C16	2545(3)	3117(2)	2037(3)	35(1)
C17	2801(3)	3566(2)	1357(3)	33(1)
C18	2340(3)	4095(1)	1460(3)	27(1)
C19	2421(3)	4670(2)	871(3)	33(1)
C20	3654(3)	4689(1)	723(3)	29(1)
C21	3939(3)	4700(2)	-263(3)	36(1)
C22	5262(3)	4707(2)	131(3)	34(1)

C23	5770(3)	4703(1)	1372(3)	30(1)
C24	7065(3)	4729(1)	2250(3)	29(1)
C25	8181(3)	4764(2)	2110(3)	35(1)
C26	9161(3)	4799(1)	3286(3)	33(1)
C27	8624(3)	4781(1)	4108(3)	28(1)
C28	9168(3)	4820(1)	5462(3)	31(1)
C29	9381(4)	3010(2)	9225(3)	66(1)
C30	7482(4)	3636(2)	8870(3)	57(1)
C31	159(4)	2660(2)	2696(4)	57(1)
C32	1165(4)	3380(2)	4353(3)	49(1)
C33	1320(3)	4709(2)	-371(3)	40(1)
C34	2327(4)	5194(2)	1642(3)	41(1)
C35	10607(3)	4885(2)	5942(3)	41(1)
C36	8626(4)	5365(2)	5829(3)	43(1)
O1A	5390(2)	4312(1)	4122(2)	35(1)
O2A	4882(2)	3764(1)	5393(2)	37(1)
C1A	4682(3)	4791(1)	5465(3)	25(1)
C2A	4271(3)	4754(2)	6392(3)	40(1)
C3A	3946(4)	5256(2)	6843(3)	46(1)
C4A	3999(4)	5789(2)	6367(3)	44(1)
C5A	4417(4)	5838(2)	5455(3)	41(1)
C6A	4751(3)	5338(2)	5012(3)	34(1)
C7A	5021(3)	4246(2)	4958(3)	27(1)
N1B	3412(3)	1711(1)	-802(2)	34(1)
C1B	2492(4)	1470(2)	-1992(3)	45(1)
C2B	1143(4)	1664(2)	-2400(4)	64(1)
C3B	366(4)	1421(2)	-3661(4)	66(1)
C4B	-1015(7)	1447(5)	-4021(10)	81(4)
C4BA	-848(12)	1710(7)	-4328(14)	128(11)
C5B	4679(3)	1430(2)	-584(3)	39(1)
C6B	5748(3)	1595(2)	581(4)	50(1)
C7B	6964(4)	1301(2)	713(4)	55(1)
C8B	8038(4)	1404(2)	1904(5)	84(2)
C9B	3495(4)	2375(2)	-845(3)	39(1)
C10B	3921(5)	2620(2)	-1780(4)	57(1)
C11B	3745(4)	3284(2)	-1872(4)	53(1)

C12B	2402(4)	3481(2)	-2268(4)	64(1)
C13B	3008(3)	1580(2)	223(3)	37(1)
C14B	2902(4)	942(2)	487(4)	54(1)
C15B	2337(4)	872(2)	1408(4)	61(1)
C16B	976(4)	1063(3)	959(5)	86(2)
Cl1C	5713(2)	2352(1)	3511(1)	80(1)
Cl2C	6379(2)	3194(1)	2085(2)	93(1)
C1C	5960(4)	3092(2)	3304(4)	64(1)

Table 3. Bond lengths [Å] and angles [°] for **7**·(C₄H₉)₄NPhCO₂·CH₂Cl₂.

S1-C1	1.722(3)	C11-C12	1.410(5)
S1-C4	1.725(4)	C11-H11	0.96
S2-C15	1.723(3)	C12-C13	1.375(4)
S2-C18	1.722(3)	C12-H12	0.96
N1-C9	1.368(4)	C13-C14	1.508(5)
N1-C6	1.375(4)	C14-C15	1.524(4)
N1-H1N	0.89(4)	C14-C31	1.531(5)
N2-C13	1.371(4)	C14-C32	1.537(5)
N2-C10	1.374(4)	C15-C16	1.345(4)
N2-H2N	0.84(4)	C16-C17	1.418(5)
N3-C23	1.372(4)	C16-H16	0.96
N3-C20	1.378(4)	C17-C18	1.347(5)
N3-H3N	0.81(3)	C17-H17	0.96
N4-C27	1.373(4)	C18-C19	1.517(5)
N4-C24	1.374(4)	C19-C20	1.509(5)
N4-H4N	0.79(3)	C19-C33	1.535(4)
C1-C2	1.352(5)	C19-C34	1.547(5)
C1-C28	1.521(5)	C20-C21	1.360(4)
C2-C3	1.415(5)	C21-C22	1.411(5)
C2-H2	0.96	C21-H21	0.96
C3-C4	1.353(5)	C22-C23	1.377(4)
C3-H3	0.96	C22-H22	0.96
C4-C5	1.528(5)	C23-C24	1.450(4)
C5-C6	1.494(5)	C24-C25	1.371(4)
C5-C29	1.536(5)	C25-C26	1.423(5)
C5-C30	1.545(5)	C25-H25	0.96
C6-C7	1.373(5)	C26-C27	1.368(4)
C7-C8	1.417(5)	C26-H26	0.96
C7-H7	0.96	C27-C28	1.505(4)
C8-C9	1.365(4)	C28-C36	1.537(5)
C8-H8	0.96	C28-C35	1.536(5)
C9-C10	1.457(4)	C29-H29A	0.96
C10-C11	1.374(5)	C29-H29B	0.96

C29-H29C	0.96	N1B-C1B	1.510(4)
C30-H30A	0.96	N1B-C13B	1.521(4)
C30-H30B	0.96	N1B-C9B	1.521(4)
C30-H30C	0.96	N1B-C5B	1.524(4)
C31-H31A	0.96	C1B-C2B	1.504(5)
C31-H31B	0.96	C1B-H1BA	0.96
C31-H31C	0.96	C1B-H1BB	0.96
C32-H32A	0.96	C2B-C3B	1.531(5)
C32-H32B	0.96	C2B-H2BA	0.96
C32-H32C	0.96	C2B-H2BB	0.96
C33-H33A	0.96	C3B-C4BA	1.468(9)
C33-H33B	0.96	C3B-C4B	1.481(8)
C33-H33C	0.96	C3B-H3BA	0.96
C34-H34A	0.96	C3B-H3BB	0.96
C34-H34B	0.96	C4B-H4BA	0.96
C34-H34C	0.96	C4B-H4BB	0.96
C35-H35A	0.96	C4B-H4BC	0.96
C35-H35B	0.96	C4BA-H4BD	0.96
C35-H35C	0.96	C4BA-H4BE	0.96
C36-H36A	0.96	C4BA-H4BF	0.96
C36-H36B	0.96	C5B-C6B	1.504(5)
C36-H36C	0.96	C5B-H5BA	0.96
O1A-C7A	1.255(3)	C5B-H5BB	0.96
O2A-C7A	1.259(4)	C6B-C7B	1.509(5)
C1A-C6A	1.381(4)	C6B-H6BA	0.96
C1A-C2A	1.385(4)	C6B-H6BB	0.96
C1A-C7A	1.507(4)	C7B-C8B	1.498(6)
C2A-C3A	1.385(5)	C7B-H7BA	0.96
C2A-H2A	0.96	C7B-H7BB	0.96
C3A-C4A	1.359(5)	C8B-H8BA	0.96
C3A-H3A	0.96	C8B-H8BB	0.96
C4A-C5A	1.376(5)	C8B-H8BC	0.96
C4A-H4A	0.96	C9B-C10B	1.513(4)
C5A-C6A	1.381(5)	C9B-H9BA	0.96
C5A-H5A	0.96	C9B-H9BB	0.96
C6A-H6A	0.96	C10B-C11B	1.528(5)

C10B-H10A	0.96	C14B-H14A	0.96
C10B-H10B	0.96	C14B-H14B	0.96
C11B-C12B	1.501(6)	C15B-C16B	1.511(6)
C11B-H11A	0.96	C15B-H15A	0.96
C11B-H11B	0.96	C15B-H15B	0.96
C12B-H12A	0.96	C16B-H16A	0.96
C12B-H12B	0.96	C16B-H16B	0.96
C12B-H12C	0.96	C16B-H16C	0.96
C13B-C14B	1.508(5)	C11C-C1C	1.749(5)
C13B-H13A	0.96	C12C-C1C	1.743(4)
C13B-H13B	0.96	C1C-H1CA	0.96
C14B-C15B	1.510(5)	C1C-H1CB	0.96
C1-S1-C4	93.19(17)	C3-C4-C5	128.1(3)
C15-S2-C18	93.00(15)	C3-C4-S1	109.4(3)
C9-N1-C6	110.6(3)	C5-C4-S1	122.5(3)
C9-N1-H1N	127(2)	C6-C5-C4	109.3(3)
C6-N1-H1N	122(2)	C6-C5-C29	109.9(3)
C13-N2-C10	110.1(3)	C4-C5-C29	108.1(3)
C13-N2-H2N	128(3)	C6-C5-C30	109.6(3)
C10-N2-H2N	122(3)	C4-C5-C30	110.7(3)
C23-N3-C20	110.1(3)	C29-C5-C30	109.3(3)
C23-N3-H3N	124(2)	N1-C6-C7	106.4(3)
C20-N3-H3N	124(2)	N1-C6-C5	121.1(3)
C27-N4-C24	110.2(3)	C7-C6-C5	132.5(3)
C27-N4-H4N	121(2)	C6-C7-C8	108.1(3)
C24-N4-H4N	127(2)	C6-C7-H7	125.5
C2-C1-C28	128.1(3)	C8-C7-H7	126.4
C2-C1-S1	109.9(3)	C9-C8-C7	107.5(3)
C28-C1-S1	122.0(2)	C9-C8-H8	125.7
C1-C2-C3	113.4(3)	C7-C8-H8	126.8
C1-C2-H2	122.4	C8-C9-N1	107.3(3)
C3-C2-H2	124.1	C8-C9-C10	130.5(3)
C4-C3-C2	114.1(3)	N1-C9-C10	122.1(3)
C4-C3-H3	122.9	N2-C10-C11	106.9(3)
C2-C3-H3	123.1	N2-C10-C9	122.3(3)

C11-C10-C9	130.6(3)	N3-C20-C19	120.3(3)
C10-C11-C12	108.1(3)	C20-C21-C22	108.4(3)
C10-C11-H11	125.5	C20-C21-H21	125.4
C12-C11-H11	126.4	C22-C21-H21	126.2
C13-C12-C11	107.5(3)	C23-C22-C21	107.5(3)
C13-C12-H12	125.5	C23-C22-H22	125.7
C11-C12-H12	127.0	C21-C22-H22	126.8
N2-C13-C12	107.4(3)	N3-C23-C22	107.0(3)
N2-C13-C14	121.1(3)	N3-C23-C24	121.4(3)
C12-C13-C14	131.2(3)	C22-C23-C24	131.5(3)
C13-C14-C15	110.0(3)	C25-C24-N4	107.5(3)
C13-C14-C31	110.2(3)	C25-C24-C23	131.4(3)
C15-C14-C31	109.7(3)	N4-C24-C23	121.1(3)
C13-C14-C32	108.5(3)	C24-C25-C26	107.1(3)
C15-C14-C32	109.9(3)	C24-C25-H25	126.0
C31-C14-C32	108.6(3)	C26-C25-H25	126.9
C16-C15-C14	129.1(3)	C27-C26-C25	108.2(3)
C16-C15-S2	110.2(2)	C27-C26-H26	126.0
C14-C15-S2	120.7(2)	C25-C26-H26	125.8
C15-C16-C17	113.0(3)	C26-C27-N4	107.0(3)
C15-C16-H16	123.1	C26-C27-C28	132.6(3)
C17-C16-H16	123.9	N4-C27-C28	120.4(3)
C18-C17-C16	114.3(3)	C27-C28-C1	109.8(3)
C18-C17-H17	122.8	C27-C28-C36	108.7(3)
C16-C17-H17	122.9	C1-C28-C36	110.8(3)
C17-C18-C19	129.2(3)	C27-C28-C35	109.5(3)
C17-C18-S2	109.5(2)	C1-C28-C35	109.4(3)
C19-C18-S2	121.3(2)	C36-C28-C35	108.6(3)
C20-C19-C18	108.7(3)	C5-C29-H29A	109.0
C20-C19-C33	109.7(3)	C5-C29-H29B	109.0
C18-C19-C33	109.1(3)	H29A-C29-H29B	109.5
C20-C19-C34	110.4(3)	C5-C29-H29C	110.5
C18-C19-C34	110.7(3)	H29A-C29-H29C	109.5
C33-C19-C34	108.3(3)	H29B-C29-H29C	109.5
C21-C20-N3	107.0(3)	C5-C30-H30A	109.6
C21-C20-C19	132.7(3)	C5-C30-H30B	109.7

H30A-C30-H30B	109.5	H36A-C36-H36B	109.5
C5-C30-H30C	109.1	C28-C36-H36C	109.0
H30A-C30-H30C	109.5	H36A-C36-H36C	109.5
H30B-C30-H30C	109.5	H36B-C36-H36C	109.5
C14-C31-H31A	109.8	C6A-C1A-C2A	118.0(3)
C14-C31-H31B	110.2	C6A-C1A-C7A	121.3(3)
H31A-C31-H31B	109.5	C2A-C1A-C7A	120.7(3)
C14-C31-H31C	108.4	C3A-C2A-C1A	120.3(3)
H31A-C31-H31C	109.5	C3A-C2A-H2A	119.7
H31B-C31-H31C	109.5	C1A-C2A-H2A	120.0
C14-C32-H32A	109.7	C4A-C3A-C2A	120.6(3)
C14-C32-H32B	109.1	C4A-C3A-H3A	119.5
H32A-C32-H32B	109.5	C2A-C3A-H3A	119.9
C14-C32-H32C	109.6	C3A-C4A-C5A	120.2(3)
H32A-C32-H32C	109.5	C3A-C4A-H4A	120.2
H32B-C32-H32C	109.5	C5A-C4A-H4A	119.6
C19-C33-H33A	109.2	C4A-C5A-C6A	119.2(3)
C19-C33-H33B	109.3	C4A-C5A-H5A	120.8
H33A-C33-H33B	109.5	C6A-C5A-H5A	119.9
C19-C33-H33C	109.8	C1A-C6A-C5A	121.7(3)
H33A-C33-H33C	109.5	C1A-C6A-H6A	119.1
H33B-C33-H33C	109.5	C5A-C6A-H6A	119.2
C19-C34-H34A	109.0	O1A-C7A-O2A	125.7(3)
C19-C34-H34B	109.7	O1A-C7A-C1A	117.3(3)
H34A-C34-H34B	109.5	O2A-C7A-C1A	117.0(3)
C19-C34-H34C	109.7	C1B-N1B-C13B	112.1(3)
H34A-C34-H34C	109.5	C1B-N1B-C9B	111.1(3)
H34B-C34-H34C	109.5	C13B-N1B-C9B	105.5(2)
C28-C35-H35A	109.1	C1B-N1B-C5B	105.9(3)
C28-C35-H35B	109.3	C13B-N1B-C5B	111.5(3)
H35A-C35-H35B	109.5	C9B-N1B-C5B	110.9(3)
C28-C35-H35C	110.0	C2B-C1B-N1B	116.7(3)
H35A-C35-H35C	109.5	C2B-C1B-H1BA	106.6
H35B-C35-H35C	109.5	N1B-C1B-H1BA	109.3
C28-C36-H36A	109.2	C2B-C1B-H1BB	108.3
C28-C36-H36B	110.2	N1B-C1B-H1BB	108.1

H1BA-C1B-H1BB	107.6	C7B-C6B-H6BA	109.6
C1B-C2B-C3B	109.9(4)	C5B-C6B-H6BB	109.3
C1B-C2B-H2BA	108.3	C7B-C6B-H6BB	108.9
C3B-C2B-H2BA	109.8	H6BA-C6B-H6BB	108.3
C1B-C2B-H2BB	110.4	C8B-C7B-C6B	114.3(4)
C3B-C2B-H2BB	109.7	C8B-C7B-H7BA	109.4
H2BA-C2B-H2BB	108.7	C6B-C7B-H7BA	109.0
C4BA-C3B-C2B	115.8(6)	C8B-C7B-H7BB	108.0
C4B-C3B-C2B	114.4(5)	C6B-C7B-H7BB	108.1
C4B-C3B-H3BA	104.3	H7BA-C7B-H7BB	107.9
C2B-C3B-H3BA	107.8	C7B-C8B-H8BA	110.6
C4B-C3B-H3BB	114.1	C7B-C8B-H8BB	108.2
C2B-C3B-H3BB	108.2	H8BA-C8B-H8BB	109.5
H3BA-C3B-H3BB	107.7	C7B-C8B-H8BC	109.6
C3B-C4B-H4BA	109.6	H8BA-C8B-H8BC	109.5
C3B-C4B-H4BB	108.7	H8BB-C8B-H8BC	109.5
H4BA-C4B-H4BB	109.5	C10B-C9B-N1B	116.0(3)
C3B-C4B-H4BC	110.1	C10B-C9B-H9BA	106.9
H4BA-C4B-H4BC	109.5	N1B-C9B-H9BA	107.1
H4BB-C4B-H4BC	109.5	C10B-C9B-H9BB	109.4
H4BD-C4B-H4BE	105.7	N1B-C9B-H9BB	109.2
H4BA-C4BA-H4BC	102.0	H9BA-C9B-H9BB	107.9
C3B-C4BA-H4BD	111.0	C9B-C10B-C11B	110.4(3)
C3B-C4BA-H4BE	105.9	C9B-C10B-H10A	107.8
H4BD-C4BA-H4BE	109.5	C11B-C10B-H10A	108.7
C3B-C4BA-H4BF	111.4	C9B-C10B-H10B	110.9
H4BD-C4BA-H4BF	109.5	C11B-C10B-H10B	110.5
H4BE-C4BA-H4BF	109.5	H10A-C10B-H10B	108.5
C6B-C5B-N1B	115.5(3)	C12B-C11B-C10B	114.4(4)
C6B-C5B-H5BA	107.6	C12B-C11B-H11A	108.9
N1B-C5B-H5BA	108.8	C10B-C11B-H11A	110.1
C6B-C5B-H5BB	107.9	C12B-C11B-H11B	107.9
N1B-C5B-H5BB	109.0	C10B-C11B-H11B	107.4
H5BA-C5B-H5BB	107.6	H11A-C11B-H11B	107.8
C5B-C6B-C7B	111.8(3)	C11B-C12B-H12A	110.6
C5B-C6B-H6BA	109.0	C11B-C12B-H12B	108.4

H12A-C12B-H12B	109.5	C14B-C15B-H15A	107.6
C11B-C12B-H12C	109.4	C16B-C15B-H15A	107.4
H12A-C12B-H12C	109.5	C14B-C15B-H15B	110.5
H12B-C12B-H12C	109.5	C16B-C15B-H15B	109.5
C14B-C13B-N1B	116.1(3)	H15A-C15B-H15B	108.0
C14B-C13B-H13A	107.3	C15B-C16B-H16A	111.2
N1B-C13B-H13A	108.3	C15B-C16B-H16B	109.7
C14B-C13B-H13B	108.4	H16A-C16B-H16B	109.5
N1B-C13B-H13B	108.6	C15B-C16B-H16C	107.5
H13A-C13B-H13B	107.8	H16A-C16B-H16C	109.5
C15B-C14B-C13B	110.8(3)	H16B-C16B-H16C	109.5
C15B-C14B-H14A	110.6	Cl2C-C1C-Cl1C	111.6(2)
C13B-C14B-H14A	108.8	Cl2C-C1C-H1CA	109.2
C15B-C14B-H14B	108.6	Cl1C-C1C-H1CA	109.2
C13B-C14B-H14B	109.5	Cl2C-C1C-H1CB	109.1
H14A-C14B-H14B	108.5	Cl1C-C1C-H1CB	109.5
C14B-C15B-C16B	113.7(4)	H1CA-C1C-H1CB	108.2

Table 4. Anisotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for $7 \cdot (\text{C}_4\text{H}_9)_4\text{NPhCO}_2 \cdot \text{CH}_2\text{Cl}_2$. The anisotropic displacement factor exponent takes the form: $-2\pi^2 [h^2 a^{*2} U^{11} + \dots + 2 h k a^* b^* U^{12}]$.

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
S1	54(1)	31(1)	29(1)	-5(1)	14(1)	-5(1)
S2	46(1)	31(1)	45(1)	6(1)	28(1)	6(1)
N1	39(2)	21(2)	28(2)	6(1)	16(1)	3(1)
N2	34(2)	18(2)	33(2)	1(1)	15(1)	-2(1)
N3	39(2)	29(2)	23(2)	3(1)	12(1)	0(1)
N4	30(2)	24(2)	34(2)	2(1)	16(1)	-2(1)
C1	26(2)	23(2)	34(2)	0(2)	8(1)	1(2)
C2	51(2)	31(2)	52(2)	2(2)	36(2)	0(2)
C3	58(2)	23(2)	57(2)	3(2)	36(2)	1(2)
C4	32(2)	30(2)	34(2)	7(2)	8(2)	1(2)
C5	43(2)	40(2)	29(2)	6(2)	10(2)	-8(2)
C6	38(2)	31(2)	25(2)	7(2)	13(2)	3(2)
C7	41(2)	40(2)	33(2)	14(2)	14(2)	11(2)
C8	51(2)	21(2)	36(2)	7(2)	25(2)	7(2)
C9	39(2)	19(2)	28(2)	2(2)	21(2)	0(2)
C10	38(2)	16(2)	28(2)	1(1)	20(2)	0(2)
C11	47(2)	19(2)	44(2)	4(2)	22(2)	-2(2)
C12	43(2)	26(2)	44(2)	-2(2)	20(2)	-12(2)
C13	35(2)	26(2)	31(2)	2(2)	16(2)	-4(2)
C14	34(2)	32(2)	37(2)	6(2)	16(2)	-2(2)
C15	27(2)	26(2)	32(2)	2(2)	11(2)	2(2)
C16	44(2)	25(2)	38(2)	1(2)	19(2)	2(2)
C17	42(2)	29(2)	35(2)	1(2)	23(2)	3(2)
C18	28(2)	27(2)	26(2)	-1(1)	9(1)	-2(2)
C19	36(2)	28(2)	30(2)	3(2)	10(2)	4(2)
C20	36(2)	21(2)	28(2)	4(2)	9(2)	-4(2)
C21	44(2)	35(2)	26(2)	3(2)	11(2)	-10(2)
C22	49(2)	28(2)	32(2)	1(2)	22(2)	-7(2)
C23	40(2)	19(2)	35(2)	0(2)	20(2)	-4(2)

C24	41(2)	20(2)	30(2)	4(2)	17(2)	3(2)
C25	46(2)	30(2)	37(2)	2(2)	25(2)	0(2)
C26	31(2)	26(2)	46(2)	1(2)	21(2)	2(2)
C27	31(2)	17(2)	37(2)	1(2)	14(2)	1(2)
C28	37(2)	20(2)	37(2)	2(2)	14(2)	1(2)
C29	59(3)	72(3)	42(2)	20(2)	-4(2)	-16(2)
C30	84(3)	57(3)	38(2)	-12(2)	31(2)	-24(2)
C31	38(2)	68(3)	59(3)	16(2)	14(2)	-13(2)
C32	53(2)	55(3)	55(2)	20(2)	38(2)	21(2)
C33	37(2)	44(2)	34(2)	10(2)	8(2)	1(2)
C34	44(2)	32(2)	45(2)	4(2)	16(2)	0(2)
C35	37(2)	33(2)	46(2)	3(2)	8(2)	-7(2)
C36	67(3)	22(2)	41(2)	-1(2)	21(2)	3(2)
O1A	42(1)	33(1)	33(1)	0(1)	19(1)	3(1)
O2A	44(2)	20(1)	43(1)	4(1)	14(1)	-2(1)
C1A	29(2)	24(2)	23(2)	-2(2)	10(1)	-3(2)
C2A	62(2)	22(2)	46(2)	8(2)	33(2)	0(2)
C3A	75(3)	36(2)	48(2)	5(2)	45(2)	5(2)
C4A	64(3)	27(2)	47(2)	-4(2)	29(2)	7(2)
C5A	66(3)	19(2)	49(2)	5(2)	35(2)	4(2)
C6A	50(2)	26(2)	34(2)	5(2)	26(2)	0(2)
C7A	26(2)	25(2)	27(2)	-1(2)	7(1)	-1(2)
N1B	56(2)	20(2)	37(2)	-3(1)	29(2)	2(1)
C1B	73(3)	27(2)	41(2)	-5(2)	30(2)	-1(2)
C2B	67(3)	58(3)	61(3)	-18(2)	19(2)	-2(3)
C3B	89(4)	41(3)	61(3)	-4(2)	22(3)	5(2)
C4B	55(6)	56(8)	113(9)	-38(6)	13(6)	12(5)
C4BA	127(16)	63(14)	118(15)	-50(12)	-29(13)	11(11)
C5B	64(3)	21(2)	50(2)	-2(2)	41(2)	3(2)
C6B	55(2)	40(2)	63(3)	-12(2)	32(2)	3(2)
C7B	70(3)	40(2)	76(3)	9(2)	50(3)	8(2)
C8B	50(3)	88(4)	115(4)	-13(3)	33(3)	-2(3)
C9B	63(3)	20(2)	45(2)	-1(2)	34(2)	4(2)
C10B	103(4)	26(2)	76(3)	4(2)	72(3)	3(2)
C11B	87(3)	25(2)	65(3)	5(2)	48(3)	1(2)
C12B	97(4)	38(3)	45(2)	6(2)	14(3)	7(2)

C13B	54(2)	30(2)	41(2)	1(2)	34(2)	1(2)
C14B	85(3)	35(2)	62(3)	10(2)	50(3)	-1(2)
C15B	76(3)	57(3)	65(3)	12(2)	46(3)	-6(2)
C16B	67(3)	114(5)	98(4)	31(3)	54(3)	-10(3)
Cl1C	137(1)	47(1)	86(1)	12(1)	75(1)	19(1)
Cl2C	133(1)	44(1)	130(1)	8(1)	82(1)	-10(1)
C1C	78(3)	43(3)	68(3)	-18(2)	26(3)	6(2)

Table 5. Hydrogen coordinates ($\times 10^4$) and isotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for $7 \cdot (\text{C}_4\text{H}_9)_4\text{NPhCO}_2 \cdot \text{CH}_2\text{Cl}_2$.

	x	y	z	U(eq)
H2	8961	3612	4897	48
H3	8504	2884	6185	51
H7	8108	1940	8638	47
H8	6097	1425	7218	41
H11	3643	1334	5485	42
H12	1537	1689	3895	44
H16	2808	2718	2050	41
H17	3273	3500	873	39
H21	3335	4701	-1086	43
H22	5732	4707	-368	41
H25	8275	4766	1357	42
H26	10046	4827	3469	39
H29A	9767	2750	8851	98
H29B	9917	3343	9545	98
H29C	9259	2809	9867	98
H30A	6683	3764	8281	86
H30B	7359	3436	9512	86
H30C	8017	3970	9190	86
H31A	-151	2447	3205	85
H31B	-457	2945	2238	85
H31C	313	2394	2157	85
H32A	1934	3575	4843	74
H32B	545	3665	3903	74
H32C	862	3161	4860	74
H33A	1366	4387	-860	60
H33B	538	4692	-272	60
H33C	1366	5071	-756	60
H34A	3015	5175	2420	62

H34B	2371	5554	1252	62
H34C	1543	5176	1736	62
H35A	10953	4544	5726	62
H35B	10809	5225	5589	62
H35C	10958	4925	6804	62
H36A	7727	5327	5538	65
H36B	8978	5405	6691	65
H36C	8830	5705	5476	65
H2A	4217	4380	6729	48
H3A	3659	5227	7483	55
H4A	3772	6135	6684	52
H5A	4430	6210	5094	49
H6A	5075	5374	4398	40
H1BA	2489	1051	-1951	53
H1BB	2785	1577	-2603	53
H2BA	1121	2084	-2419	77
H2BB	795	1529	-1847	77
H3BA	545	1010	-3654	79
H3BB	649	1606	-4220	79
H4BA	-1438	1289	-4818	122
H4BB	-1227	1220	-3462	122
H4BC	-1276	1845	-4011	122
H4BD	-1279	1535	-5108	192
H4BE	-1335	1654	-3855	192
H4BF	-742	2121	-4420	192
H5BA	4917	1537	-1231	47
H5BB	4590	1012	-598	47
H6BA	5535	1484	1241	59
H6BB	5865	2011	605	59
H7BA	7196	1433	76	66
H7BB	6816	887	619	66
H8BA	8781	1204	1939	126
H8BB	8200	1817	1995	126
H8BC	7816	1267	2543	126
H9BA	4098	2499	-71	47
H9BB	2693	2540	-959	47

H10A	3405	2449	-2544	68
H10B	4784	2520	-1593	68
H11A	4085	3442	-2413	64
H11B	4217	3444	-1085	64
H12A	2353	3901	-2285	96
H12B	1927	3330	-3062	96
H12C	2061	3332	-1720	96
H13A	2196	1754	33	44
H13B	3594	1762	945	44
H14A	3725	768	773	65
H14B	2369	746	-241	65
H15A	2812	1113	2088	73
H15B	2411	474	1682	73
H16A	660	1024	1577	130
H16B	902	1463	698	130
H16C	497	817	288	130
H1CA	6619	3240	4022	77
H1CB	5205	3309	3167	77
H3N	4850(30)	4621(14)	2400(30)	25(9)
H1N	5840(30)	3122(17)	6460(30)	38(10)
H4N	6890(30)	4663(15)	3790(30)	33(10)
H2N	3880(40)	3010(20)	5050(30)	60(13)

Table 6. Torsion angles [°] for **7**·(C₄H₉)₄NPhCO₂·CH₂Cl₂.

C4-S1-C1-C2	2.2(3)	C8-C9-C10-C11	1.0(5)
C4-S1-C1-C28	-176.3(3)	N1-C9-C10-C11	-174.9(3)
C28-C1-C2-C3	176.6(3)	N2-C10-C11-C12	0.6(3)
S1-C1-C2-C3	-1.8(4)	C9-C10-C11-C12	177.2(3)
C1-C2-C3-C4	0.3(5)	C10-C11-C12-C13	-0.4(4)
C2-C3-C4-C5	-177.4(3)	C10-N2-C13-C12	0.4(3)
C2-C3-C4-S1	1.4(4)	C10-N2-C13-C14	174.7(3)
C1-S1-C4-C3	-2.0(3)	C11-C12-C13-N2	0.0(4)
C1-S1-C4-C5	176.8(3)	C11-C12-C13-C14	-173.5(3)
C3-C4-C5-C6	-37.9(5)	N2-C13-C14-C15	59.4(4)
S1-C4-C5-C6	143.5(3)	C12-C13-C14-C15	-127.8(4)
C3-C4-C5-C29	81.6(4)	N2-C13-C14-C31	-179.5(3)
S1-C4-C5-C29	-97.0(4)	C12-C13-C14-C31	-6.8(5)
C3-C4-C5-C30	-158.7(4)	N2-C13-C14-C32	-60.8(4)
S1-C4-C5-C30	22.7(4)	C12-C13-C14-C32	112.0(4)
C9-N1-C6-C7	0.0(3)	C13-C14-C15-C16	38.5(5)
C9-N1-C6-C5	-178.4(3)	C31-C14-C15-C16	-82.8(4)
C4-C5-C6-N1	-57.3(4)	C32-C14-C15-C16	157.9(3)
C29-C5-C6-N1	-175.7(3)	C13-C14-C15-S2	-143.7(2)
C30-C5-C6-N1	64.2(4)	C31-C14-C15-S2	95.0(3)
C4-C5-C6-C7	124.8(4)	C32-C14-C15-S2	-24.3(4)
C29-C5-C6-C7	6.3(5)	C18-S2-C15-C16	-1.0(3)
C30-C5-C6-C7	-113.7(4)	C18-S2-C15-C14	-179.2(3)
N1-C6-C7-C8	0.2(4)	C14-C15-C16-C17	178.7(3)
C5-C6-C7-C8	178.3(3)	S2-C15-C16-C17	0.7(4)
C6-C7-C8-C9	-0.3(4)	C15-C16-C17-C18	0.1(4)
C7-C8-C9-N1	0.3(3)	C16-C17-C18-C19	-179.7(3)
C7-C8-C9-C10	-176.0(3)	C16-C17-C18-S2	-0.8(4)
C6-N1-C9-C8	-0.2(3)	C15-S2-C18-C17	1.0(3)
C6-N1-C9-C10	176.5(3)	C15-S2-C18-C19	-180.0(3)
C13-N2-C10-C11	-0.6(3)	C17-C18-C19-C20	-32.7(5)
C13-N2-C10-C9	-177.6(3)	S2-C18-C19-C20	148.5(2)
C8-C9-C10-N2	177.2(3)	C17-C18-C19-C33	86.9(4)
N1-C9-C10-N2	1.3(4)	S2-C18-C19-C33	-91.9(3)

C17-C18-C19-C34	-154.1(3)	C2-C1-C28-C27	38.7(5)
S2-C18-C19-C34	27.1(4)	S1-C1-C28-C27	-143.2(2)
C23-N3-C20-C21	-1.1(4)	C2-C1-C28-C36	158.7(4)
C23-N3-C20-C19	178.9(3)	S1-C1-C28-C36	-23.1(4)
C18-C19-C20-C21	115.1(4)	C2-C1-C28-C35	-81.6(4)
C33-C19-C20-C21	-4.1(5)	S1-C1-C28-C35	96.6(3)
C34-C19-C20-C21	-123.3(4)	C6A-C1A-C2A-C3A	-0.1(5)
C18-C19-C20-N3	-64.9(4)	C7A-C1A-C2A-C3A	-179.2(3)
C33-C19-C20-N3	175.9(3)	C1A-C2A-C3A-C4A	1.4(6)
C34-C19-C20-N3	56.7(4)	C2A-C3A-C4A-C5A	-2.0(6)
N3-C20-C21-C22	0.9(4)	C3A-C4A-C5A-C6A	1.4(6)
C19-C20-C21-C22	-179.1(3)	C2A-C1A-C6A-C5A	-0.5(5)
C20-C21-C22-C23	-0.5(4)	C7A-C1A-C6A-C5A	178.6(3)
C20-N3-C23-C22	0.8(4)	C4A-C5A-C6A-C1A	-0.2(6)
C20-N3-C23-C24	178.3(3)	C6A-C1A-C7A-O1A	1.2(4)
C21-C22-C23-N3	-0.2(4)	C2A-C1A-C7A-O1A	-179.8(3)
C21-C22-C23-C24	-177.4(3)	C6A-C1A-C7A-O2A	-177.3(3)
C27-N4-C24-C25	0.3(4)	C2A-C1A-C7A-O2A	1.8(4)
C27-N4-C24-C23	-178.0(3)	C13B-N1B-C1B-C2B	56.0(4)
N3-C23-C24-C25	-176.8(3)	C9B-N1B-C1B-C2B	-61.8(4)
C22-C23-C24-C25	0.0(6)	C5B-N1B-C1B-C2B	177.8(3)
N3-C23-C24-N4	1.1(5)	N1B-C1B-C2B-C3B	175.4(3)
C22-C23-C24-N4	177.9(3)	C1B-C2B-C3B-C4BA	-161.9(11)
N4-C24-C25-C26	-0.4(4)	C1B-C2B-C3B-C4B	164.6(7)
C23-C24-C25-C26	177.7(3)	C1B-N1B-C5B-C6B	-178.5(3)
C24-C25-C26-C27	0.3(4)	C13B-N1B-C5B-C6B	-56.3(4)
C25-C26-C27-N4	-0.1(4)	C9B-N1B-C5B-C6B	60.9(4)
C25-C26-C27-C28	-177.8(3)	N1B-C5B-C6B-C7B	-179.5(3)
C24-N4-C27-C26	-0.1(4)	C5B-C6B-C7B-C8B	-174.7(4)
C24-N4-C27-C28	177.9(3)	C1B-N1B-C9B-C10B	-60.0(4)
C26-C27-C28-C1	-120.9(4)	C13B-N1B-C9B-C10B	178.3(3)
N4-C27-C28-C1	61.7(4)	C5B-N1B-C9B-C10B	57.5(4)
C26-C27-C28-C36	117.8(4)	N1B-C9B-C10B-C11B	170.3(3)
N4-C27-C28-C36	-59.6(4)	C9B-C10B-C11B-C12B	-62.0(5)
C26-C27-C28-C35	-0.8(5)	C1B-N1B-C13B-C14B	61.1(4)
N4-C27-C28-C35	-178.2(3)	C9B-N1B-C13B-C14B	-177.8(3)

C5B-N1B-C13B-C14B	-57.4(4)
N1B-C13B-C14B-C15B	-172.8(3)
C13B-C14B-C15B-C16B	66.9(5)

Table 7. Hydrogen bonds for **7**·(C₄H₉)₄NPhCO₂·CH₂Cl₂ [Å and °].

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)
N1-H1N...O2A	0.89(4)	1.98(4)	2.847(4)	165(3)
N2-H2N...O2A	0.84(4)	2.02(4)	2.848(4)	172(4)
N3-H3N...O1A	0.81(3)	2.05(3)	2.840(3)	166(3)
N4-H4N...O1A	0.79(3)	2.09(3)	2.854(4)	161(3)

Figure 1. View of the macrocycle in **7**·(C₄H₉)₄NPhCO₂·CH₂Cl₂ showing the atom labeling scheme. Displacement ellipsoids are scaled to the 50% probability level. The methyl hydrogen atoms have been removed for clarity.

Figure 2. View illustrating the H-bonding interaction between the benzoate anion and the macrocycle in $7 \cdot (\text{C}_4\text{H}_9)_4\text{N PhCO}_2\text{-CH}_2\text{Cl}_2$. Displacement ellipsoids are scaled to the 50% probability level. Most hydrogen atoms have been removed for clarity. Dashed lines are indicative of H-bonding interactions. The geometry of these interactions are listed in Table 7.

Figure 3. View of the cation in $7 \cdot (\text{C}_4\text{H}_9)_4\text{NPhCO}_2 \cdot \text{CH}_2\text{Cl}_2$ showing the atom labeling scheme. Displacement ellipsoids are scaled to the 50% probability level. The hydrogen atoms have been removed for clarity. The methyl carbon on one n-butyl group was found to be disordered about two positions represented by atoms labeled C4b (58(2)% occupancy) and C4ba (42(2)% occupancy).

Figure 4. Unit cell packing diagram for $7 \cdot (\text{C}_4\text{H}_9)_4\text{NPhCO}_2 \cdot \text{CH}_2\text{Cl}_2$. The view is approximately down the *c* axis. Dashed lines are indicative of H-bonding interactions, which are listed in Table 7.

Crystallographic Material for $7 \cdot (\text{C}_4\text{H}_9)_4\text{NCH}_3\text{CO}_2 \cdot \text{CH}_2\text{Cl}_2$

- X-ray Experimental.
- Table 1. Crystallographic Data for $7 \cdot (\text{C}_4\text{H}_9)_4\text{NCH}_3\text{CO}_2 \cdot \text{CH}_2\text{Cl}_2$.
- Table 2. Fractional coordinates and equivalent isotropic thermal parameters ($\text{\AA}^2$) for the non-hydrogen atoms of $7 \cdot (\text{C}_4\text{H}_9)_4\text{NCH}_3\text{CO}_2 \cdot \text{CH}_2\text{Cl}_2$.
- Table 3. Bond Lengths ($\text{\AA}$) and Angles ($^\circ$) for the non-hydrogen atoms of $7 \cdot (\text{C}_4\text{H}_9)_4\text{NCH}_3\text{CO}_2 \cdot \text{CH}_2\text{Cl}_2$.
- Table 4. Anisotropic thermal parameters for the non-hydrogen atoms of $7 \cdot (\text{C}_4\text{H}_9)_4\text{NCH}_3\text{CO}_2 \cdot \text{CH}_2\text{Cl}_2$.
- Table 5. Fractional coordinates and isotropic thermal parameters ($\text{\AA}^2$) for the hydrogen atoms of $7 \cdot (\text{C}_4\text{H}_9)_4\text{NCH}_3\text{CO}_2 \cdot \text{CH}_2\text{Cl}_2$.
- Table 6. Torsion Angles ($^\circ$) for the non-hydrogen atoms of $7 \cdot (\text{C}_4\text{H}_9)_4\text{NCH}_3\text{CO}_2 \cdot \text{CH}_2\text{Cl}_2$.
- Table 7. Hydrogen Bond Lengths ($\text{\AA}$) and Angles ($^\circ$) for $7 \cdot (\text{C}_4\text{H}_9)_4\text{NCH}_3\text{CO}_2 \cdot \text{CH}_2\text{Cl}_2$.
- Figure 1. View of the macrocycle acetate complex in $7 \cdot (\text{C}_4\text{H}_9)_4\text{NCH}_3\text{CO}_2 \cdot \text{CH}_2\text{Cl}_2$ showing the atom labeling scheme.
- Figure 2. View of the tetrabutylammonium cation in $7 \cdot (\text{C}_4\text{H}_9)_4\text{NCH}_3\text{CO}_2 \cdot \text{CH}_2\text{Cl}_2$ showing the atom labeling scheme.
- Figure 3. Unit cell packing diagram for $7 \cdot (\text{C}_4\text{H}_9)_4\text{NCH}_3\text{CO}_2 \cdot \text{CH}_2\text{Cl}_2$.

X-ray Experimental for **7**·(C₄H₉)₄NCH₃CO₂·CH₂Cl₂: C₅₅H₈₁Cl₂N₅O₂S₂, crystals grew as colorless plates by slow evaporation of a dichloromethane solution containing **7** and tetrabutylammonium acetate (1:1 molar ratio). The data crystal was cut from a larger plate and had approximate dimensions; 0.27 × 0.26 × 0.10 mm. The data were collected on a Nonius Kappa CCD diffractometer using a graphite monochromator with MoK α radiation (λ = 0.71073 Å). A total of 461 frames of data were collected using ω -scans with a scan range of 1° and a counting time of 151 seconds per frame. The data were collected at 153 K using an Oxford Cryostream low temperature device. Details of crystal data, data collection and structure refinement are listed in Table 1. Data reduction were performed using DENZO-SMN.¹ The structure was solved by direct methods using SIR97² and refined by full-matrix least-squares on F² with anisotropic displacement parameters for the non-H atoms using SHELXL-97.³ The hydrogen atoms on carbon were calculated in ideal positions with isotropic displacement parameters set to 1.2 × U_{eq} of the attached atom (1.5 × U_{eq} for methyl hydrogen atoms). The hydrogen atoms on the pyrrole nitrogen atoms were observed in a ΔF map and refined with isotropic displacement parameters.

The complex crystallized along with a molecule of methylene chloride. The methylene chloride was disordered by rotation of the chlorine atoms about a circle below the methylene carbon atom, C1C. Six distinct Cl atom positions could be isolated. These six atoms paired up with C1C to form three geometrically reasonable orientations for the molecule. After the remainder of the structure was refined with hydrogen atoms included, the disorder model was attempted. Initially, all Cl atoms were assigned a common isotropic displacement parameter. This parameter was refined as a free variable. The Cl-C bond lengths and the Cl-C-Cl bond angles were restrained to be equivalent while refining individual site occupancy factors for Cl.

The site occupancy factors were subsequently assigned and fixed so that the sum of all chlorine atoms equaled two. In addition, for pairs of chlorine atoms that formed a geometrically reasonable molecule, site occupancy factors were required to be equal. No hydrogen atoms for the methylene chloride molecule were included in the final refinement model.

In addition to the disorder of the methylene chloride molecule, one of the n-butyl groups of the cation was found to be partially disordered (Figure 2). The disorder affected the two atoms nearest the nitrogen atom, C13b and C14b, and C13c and C14c. The site occupancy factors were estimated by requiring the sum of the site occupancies of the paired atoms to equal unity while refining a common isotropic displacement parameter. The site occupancy factor refined to 59(2)% for C13b and C14b. Hydrogen atoms were calculated for these atoms.

The function, $\sum w(|F_o|^2 - |F_c|^2)^2$, was minimized, where $w = 1/[(\sigma(F_o))^2 + (0.02 \cdot P)^2]$ and $P = (|F_o|^2 + 2|F_c|^2)/3$. $R_w(F^2)$ refined to 0.142, with $R(F)$ equal to 0.0754 and a goodness of fit, S , = 1.72. Definitions used for calculating $R(F)$, $R_w(F^2)$ and the goodness of fit, S , are given below.⁴ The data were corrected for secondary extinction effects. The correction takes the form: $F_{corr} = kF_c/[1 + (2.0(5) \times 10^{-6}) \cdot F_c^2 \lambda^3/(\sin 2\theta)]^{0.25}$ where k is the overall scale factor. Neutral atom scattering factors and values used to calculate the linear absorption coefficient are from the International Tables for X-ray Crystallography (1992).⁵ All figures were generated using SHELXTL/PC.⁶ Tables of positional and thermal parameters, bond lengths and angles, torsion angles, H-bonding interactions, figures and lists of observed and calculated structure factors are located in tables 1 through 8.

References

1. DENZO-SMN. (1997). Z. Otwinowski and W. Minor, Methods in Enzymology, **276**: Macromolecular Crystallography, part A, 307 – 326, C. W. Carter, Jr. and R. M. Sweets, Editors, Academic Press.
2. SIR97. (1999). A program for crystal structure solution. Altomare A., Burla M.C., Camalli M., Cascarano G.L., Giacovazzo C. , Guagliardi A., Moliterni A.G.G., Polidori G., Spagna R. J. Appl. Cryst. 32, 115-119.
3. Sheldrick, G. M. (1994). SHELXL97. Program for the Refinement of Crystal Structures. University of Gottingen, Germany.
4. $R_w(F^2) = \{ \sum w(|F_o|^2 - |F_c|^2)^2 / \sum w(|F_o|^4) \}^{1/2}$ where w is the weight given each reflection.
 $R(F) = \sum (|F_o| - |F_c|) / \sum |F_o|$ for reflections with $F_o > 4(\sigma(F_o))$.
 $S = [\sum w(|F_o|^2 - |F_c|^2)^2 / (n - p)]^{1/2}$, where n is the number of reflections and p is the number of refined parameters.
5. International Tables for X-ray Crystallography (1992). Vol. C, Tables 4.2.6.8 and 6.1.1.4, A. J. C. Wilson, editor, Boston: Kluwer Academic Press.
6. Sheldrick, G. M. (1994). SHELXTL/PC (Version 5.03). Siemens Analytical X-ray Instruments, Inc., Madison, Wisconsin, USA.

Table 1. Crystal data and structure refinement for **7**·(C₄H₉)₄NCH₃CO₂·CH₂Cl₂.

Empirical formula	C ₅₅ H ₈₁ Cl ₂ N ₅ O ₂ S ₂	
Formula weight	979.27	
Temperature	153(2) K	
Wavelength	0.71073 Å	
Crystal system	Triclinic	
Space group	<i>P</i> -1	
Unit cell dimensions	a = 12.0220(2) Å	α = 70.8010(9)°.
	b = 14.4610(2) Å	β = 88.9990(8)°.
	c = 17.7050(3) Å	γ = 72.4930(8)°.
Volume	2760.82(8) Å ³	
Z	2	
Density (calculated)	1.178 g cm ⁻³	
Absorption coefficient	0.237 mm ⁻¹	
F(000)	1056	
Crystal size	0.27 × 0.26 × 0.10 mm	
Theta range for data collection	2.96 to 27.44°.	
Index ranges	-15 ≤ h ≤ 15, -18 ≤ k ≤ 18, -22 ≤ l ≤ 20	
Reflections collected	19709	
Independent reflections	12485 [R(int) = 0.0431]	
Completeness to theta = 27.44°	99.0 %	
Absorption correction	None	
Refinement method	Full-matrix least-squares on F ²	
Data / restraints / parameters	12485 / 64 / 666	
Goodness-of-fit on F ²	1.607	
Final R indices [I > 2σ(I)]	R1 = 0.0754, wR2 = 0.1271	
R indices (all data)	R1 = 0.1929, wR2 = 0.1416	
Extinction coefficient	2.0(5) × 10 ⁻⁶	
Largest diff. peak and hole	0.610 and -0.581 e.Å ⁻³	

Table 2. Atomic coordinates ($\times 10^4$) and equivalent isotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for $7 \cdot (\text{C}_4\text{H}_9)_4\text{NCH}_3\text{CO}_2 \cdot \text{CH}_2\text{Cl}_2$. U(eq) is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U(eq)
S1	10491(1)	3546(1)	6134(1)	53(1)
S2	6515(1)	300(1)	8475(1)	44(1)
N1	11724(3)	274(2)	7594(2)	37(1)
N2	10267(3)	-931(2)	8428(2)	36(1)
N3	6391(2)	3184(2)	8642(2)	35(1)
N4	7821(2)	4428(2)	7728(2)	39(1)
C1	10029(3)	4105(2)	6853(2)	44(1)
C2	10668(3)	3515(3)	7559(2)	49(1)
C3	11522(3)	2604(3)	7535(2)	48(1)
C4	11538(3)	2507(2)	6791(2)	39(1)
C5	12314(3)	1630(2)	6540(2)	42(1)
C6	12604(3)	649(3)	7249(2)	38(1)
C7	13627(3)	-34(3)	7675(2)	45(1)
C8	13368(3)	-828(3)	8282(2)	44(1)
C9	12176(3)	-637(2)	8236(2)	36(1)
C10	11440(3)	-1213(2)	8668(2)	35(1)
C11	11685(3)	-2116(2)	9302(2)	46(1)
C12	10644(3)	-2377(2)	9444(2)	49(1)
C13	9775(3)	-1638(2)	8900(2)	40(1)
C14	8492(3)	-1488(2)	8761(2)	43(1)
C15	7788(2)	-577(2)	9008(2)	35(1)
C16	8018(3)	-341(2)	9653(2)	43(1)
C17	7179(3)	546(2)	9715(2)	41(1)
C18	6304(3)	997(2)	9118(2)	32(1)
C19	5246(3)	1933(2)	9010(2)	38(1)
C20	5615(3)	2790(2)	9117(2)	35(1)
C21	5246(3)	3389(2)	9595(2)	44(1)
C22	5816(3)	4148(2)	9399(2)	43(1)

C23	6531(3)	4011(2)	8809(2)	34(1)
C24	7258(3)	4600(2)	8378(2)	37(1)
C25	7499(3)	5432(2)	8461(2)	49(1)
C26	8205(3)	5765(2)	7847(2)	55(1)
C27	8400(3)	5131(2)	7397(2)	43(1)
C28	9043(3)	5120(3)	6666(2)	52(1)
C29	11660(3)	1516(3)	5847(2)	53(1)
C30	13445(3)	1864(3)	6246(2)	58(1)
C31	8180(3)	-1294(3)	7868(2)	61(1)
C32	8209(3)	-2463(2)	9280(2)	68(1)
C33	4607(3)	2319(2)	8166(2)	55(1)
C34	4384(3)	1637(3)	9628(2)	58(1)
C35	8182(3)	5276(3)	5965(2)	62(1)
C36	9588(3)	6014(3)	6416(2)	76(1)
O1A	9226(2)	1153(2)	7404(1)	41(1)
O2A	7851(2)	2547(2)	7499(1)	42(1)
C1A	8766(3)	1788(3)	7768(2)	40(1)
C2A	9356(3)	1588(2)	8581(2)	49(1)
N1B	6517(3)	3190(3)	2812(2)	91(1)
C1B	6776(4)	3741(4)	1971(3)	97(2)
C2B	7760(4)	4174(4)	1878(3)	136(2)
C3B	7832(4)	4739(3)	946(3)	102(2)
C4B	8879(5)	5035(4)	828(3)	132(2)
C5B	5527(4)	2788(3)	2737(3)	79(1)
C6B	5092(4)	2261(3)	3497(2)	78(1)
C7B	4200(4)	1801(3)	3335(3)	85(1)
C8B	3720(5)	1259(4)	4075(3)	126(2)
C9B	6276(6)	3813(5)	3349(3)	139(2)
C10B	5297(5)	4741(4)	3045(3)	115(2)
C11B	4937(5)	5343(4)	3679(2)	112(2)
C12B	5878(6)	5664(4)	3966(3)	155(3)
C13B	7625(6)	2500(6)	3428(4)	89(3)
C14B	8297(8)	1662(6)	3139(6)	95(3)
C13C	7532(8)	2052(7)	3074(7)	61(3)
C14C	8593(7)	2059(6)	3446(6)	61(3)
C15B	9365(4)	916(3)	3745(2)	74(1)

C16B	10326(5)	1141(4)	4081(3)	121(2)
C11C	7948(3)	2853(2)	5370(2)	107(1)
C12C	6500(2)	1518(3)	5586(1)	101(1)
C1C	7325(5)	1941(3)	6085(3)	112(2)
C11D	5965(8)	2546(15)	5595(9)	151(6)
C11E	7301(12)	3091(6)	5367(6)	214(6)
C12E	7081(13)	1095(8)	5637(8)	259(8)
C12D	8438(13)	1356(18)	5652(12)	220(9)

Table 3. Bond lengths [Å] and angles [°] for **7**·(C₄H₉)₄NCH₃CO₂·CH₂Cl₂.

S1-C4	1.719(3)	C11-C12	1.408(4)
S1-C1	1.722(3)	C11-H11	0.96
S2-C15	1.715(3)	C12-C13	1.359(4)
S2-C18	1.723(3)	C12-H12	0.96
N1-C6	1.375(4)	C13-C14	1.505(4)
N1-C9	1.389(4)	C14-C15	1.528(4)
N1-H1N	0.81(3)	C14-C32	1.541(4)
N2-C13	1.370(4)	C14-C31	1.544(4)
N2-C10	1.377(4)	C15-C16	1.351(4)
N2-H2N	0.84(3)	C16-C17	1.409(4)
N3-C20	1.371(4)	C16-H16	0.96
N3-C23	1.379(4)	C17-C18	1.350(4)
N3-H3N	0.82(3)	C17-H17	0.96
N4-C27	1.371(4)	C18-C19	1.513(4)
N4-C24	1.381(4)	C19-C20	1.503(4)
N4-H4N	0.82(2)	C19-C33	1.539(4)
C1-C2	1.348(4)	C19-C34	1.541(4)
C1-C28	1.523(4)	C20-C21	1.379(4)
C2-C3	1.418(4)	C21-C22	1.410(4)
C2-H2	0.96	C21-H21	0.96
C3-C4	1.370(4)	C22-C23	1.369(4)
C3-H3	0.96	C22-H22	0.96
C4-C5	1.523(4)	C23-C24	1.438(4)
C5-C6	1.502(4)	C24-C25	1.370(4)
C5-C30	1.539(4)	C25-C26	1.416(4)
C5-C29	1.547(4)	C25-H25	0.96
C6-C7	1.367(4)	C26-C27	1.369(4)
C7-C8	1.401(4)	C26-H26	0.96
C7-H7	0.96	C27-C28	1.498(4)
C8-C9	1.373(4)	C28-C35	1.544(4)
C8-H8	0.96	C28-C36	1.556(4)
C9-C10	1.437(4)	C29-H29A	0.96
C10-C11	1.367(4)	C29-H29B	0.96

C29-H29C	0.96	C2B-C3B	1.596(6)
C30-H30A	0.96	C2B-H2BA	0.96
C30-H30B	0.96	C2B-H2BB	0.96
C30-H30C	0.96	C3B-C4B	1.438(6)
C31-H31A	0.96	C3B-H3BA	0.96
C31-H31B	0.96	C3B-H3BB	0.96
C31-H31C	0.96	C4B-H4BA	0.96
C32-H32A	0.96	C4B-H4BB	0.96
C32-H32B	0.96	C4B-H4BC	0.96
C32-H32C	0.96	C5B-C6B	1.485(5)
C33-H33A	0.96	C5B-H5BA	0.96
C33-H33B	0.96	C5B-H5BB	0.96
C33-H33C	0.96	C6B-C7B	1.496(5)
C34-H34A	0.96	C6B-H6BA	0.96
C34-H34B	0.96	C6B-H6BB	0.96
C34-H34C	0.96	C7B-C8B	1.495(5)
C35-H35A	0.96	C7B-H7BA	0.96
C35-H35B	0.96	C7B-H7BB	0.96
C35-H35C	0.96	C8B-H8BA	0.96
C36-H36A	0.96	C8B-H8BB	0.96
C36-H36B	0.96	C8B-H8BC	0.96
C36-H36C	0.96	C9B-C10B	1.440(7)
O1A-C1A	1.279(3)	C9B-H9BA	0.96
O2A-C1A	1.261(4)	C9B-H9BB	0.96
C1A-C2A	1.516(4)	C10B-C11B	1.613(6)
C2A-H2AC	0.96	C10B-H10A	0.96
C2A-H2AA	0.96	C10B-H10B	0.96
C2A-H2AB	0.96	C11B-C12B	1.497(6)
N1B-C9B	1.483(6)	C11B-H11A	0.96
N1B-C5B	1.498(5)	C11B-H11B	0.96
N1B-C1B	1.517(5)	C12B-H12A	0.96
N1B-C13B	1.580(7)	C12B-H12B	0.96
N1B-C13C	1.649(8)	C12B-H12C	0.96
C1B-C2B	1.481(6)	C13B-C14B	1.478(7)
C1B-H1BA	0.96	C13B-H13A	0.96
C1B-H1BB	0.96	C13B-H13B	0.96

C14B-C15B	1.555(7)	Cl1C-Cl1E	0.750(14)
C14B-H14A	0.96	Cl1C-C1C	1.836(5)
C14B-H14B	0.96	Cl1C-Cl2D	1.95(2)
C13C-C14C	1.450(8)	Cl2C-Cl2E	0.762(14)
C13C-H13C	0.96	Cl2C-Cl1D	1.437(17)
C13C-H13D	0.96	Cl2C-C1C	1.700(4)
C14C-C15B	1.547(8)	Cl2C-Cl2D	2.272(17)
C14C-H14C	0.96	C1C-Cl2D	1.675(8)
C14C-H14D	0.96	C1C-Cl1D	1.697(8)
C15B-C16B	1.474(5)	C1C-Cl1E	1.722(7)
C15B-H15A	0.96	C1C-Cl2E	1.754(7)
C15B-H15B	0.96	Cl1D-Cl1E	1.972(19)
C16B-H16C	0.96	Cl1D-Cl2E	2.102(18)
C16B-H16A	0.96	Cl1E-Cl2D	2.36(2)
C16B-H16B	0.96	Cl2E-Cl2D	1.78(2)
C4-S1-C1	93.46(16)	C4-C3-C2	113.0(3)
C15-S2-C18	93.22(14)	C4-C3-H3	122.9
C6-N1-C9	110.9(3)	C2-C3-H3	124.1
C6-N1-H1N	126(2)	C3-C4-C5	127.9(3)
C9-N1-H1N	123(2)	C3-C4-S1	109.6(2)
C13-N2-C10	110.5(3)	C5-C4-S1	122.4(2)
C13-N2-H2N	123(2)	C6-C5-C4	108.8(3)
C10-N2-H2N	124(2)	C6-C5-C30	110.1(3)
C20-N3-C23	111.1(3)	C4-C5-C30	109.9(2)
C20-N3-H3N	125(2)	C6-C5-C29	109.7(2)
C23-N3-H3N	124(2)	C4-C5-C29	109.7(3)
C27-N4-C24	111.3(3)	C30-C5-C29	108.7(2)
C27-N4-H4N	126.7(17)	C7-C6-N1	106.2(3)
C24-N4-H4N	122.0(17)	C7-C6-C5	133.8(3)
C2-C1-C28	128.1(3)	N1-C6-C5	120.0(3)
C2-C1-S1	109.7(3)	C6-C7-C8	108.7(3)
C28-C1-S1	122.2(3)	C6-C7-H7	125.1
C1-C2-C3	114.2(3)	C8-C7-H7	126.2
C1-C2-H2	122.1	C9-C8-C7	108.6(3)
C3-C2-H2	123.6	C9-C8-H8	125.2

C7-C8-H8	126.2	C18-C19-C33	110.8(2)
C8-C9-N1	105.6(3)	C20-C19-C34	110.2(3)
C8-C9-C10	132.6(3)	C18-C19-C34	109.1(2)
N1-C9-C10	121.6(3)	C33-C19-C34	108.0(3)
C11-C10-N2	106.4(3)	N3-C20-C21	106.2(3)
C11-C10-C9	131.9(3)	N3-C20-C19	121.4(3)
N2-C10-C9	121.5(3)	C21-C20-C19	132.2(3)
C10-C11-C12	108.0(3)	C20-C21-C22	108.1(3)
C10-C11-H11	125.5	C20-C21-H21	125.5
C12-C11-H11	126.5	C22-C21-H21	126.4
C13-C12-C11	108.3(3)	C23-C22-C21	108.3(3)
C13-C12-H12	124.8	C23-C22-H22	125.0
C11-C12-H12	126.8	C21-C22-H22	126.7
C12-C13-N2	106.8(3)	C22-C23-N3	106.3(3)
C12-C13-C14	132.8(3)	C22-C23-C24	130.4(3)
N2-C13-C14	120.4(3)	N3-C23-C24	123.1(3)
C13-C14-C15	108.6(2)	C25-C24-N4	106.0(3)
C13-C14-C32	109.7(3)	C25-C24-C23	131.1(3)
C15-C14-C32	109.0(2)	N4-C24-C23	122.9(3)
C13-C14-C31	109.6(3)	C24-C25-C26	108.2(3)
C15-C14-C31	111.1(3)	C24-C25-H25	125.4
C32-C14-C31	108.9(3)	C26-C25-H25	126.4
C16-C15-C14	127.9(3)	C27-C26-C25	108.2(3)
C16-C15-S2	109.8(2)	C27-C26-H26	125.6
C14-C15-S2	122.3(2)	C25-C26-H26	126.2
C15-C16-C17	113.6(3)	C26-C27-N4	106.3(3)
C15-C16-H16	122.4	C26-C27-C28	132.7(3)
C17-C16-H16	124.0	N4-C27-C28	121.0(3)
C18-C17-C16	113.9(3)	C27-C28-C1	109.7(3)
C18-C17-H17	122.5	C27-C28-C35	109.6(3)
C16-C17-H17	123.5	C1-C28-C35	110.9(3)
C17-C18-C19	128.1(3)	C27-C28-C36	109.8(3)
C17-C18-S2	109.4(2)	C1-C28-C36	108.4(3)
C19-C18-S2	122.4(2)	C35-C28-C36	108.5(3)
C20-C19-C18	110.2(2)	C5-C29-H29A	109.8
C20-C19-C33	108.5(2)	C5-C29-H29B	109.9

H29A-C29-H29B	109.5	H35A-C35-H35B	109.5
C5-C29-H29C	108.7	C28-C35-H35C	109.7
H29A-C29-H29C	109.5	H35A-C35-H35C	109.5
H29B-C29-H29C	109.5	H35B-C35-H35C	109.5
C5-C30-H30A	109.9	C28-C36-H36A	108.6
C5-C30-H30B	108.2	C28-C36-H36B	110.6
H30A-C30-H30B	109.5	H36A-C36-H36B	109.5
C5-C30-H30C	110.3	C28-C36-H36C	109.2
H30A-C30-H30C	109.5	H36A-C36-H36C	109.5
H30B-C30-H30C	109.5	H36B-C36-H36C	109.5
C14-C31-H31A	109.0	O2A-C1A-O1A	124.1(3)
C14-C31-H31B	110.1	O2A-C1A-C2A	118.9(3)
H31A-C31-H31B	109.5	O1A-C1A-C2A	117.1(3)
C14-C31-H31C	109.3	C1A-C2A-H2AC	110.0
H31A-C31-H31C	109.5	C1A-C2A-H2AA	109.3
H31B-C31-H31C	109.5	H2AC-C2A-H2AA	110.1
C14-C32-H32A	109.2	C1A-C2A-H2AB	109.4
C14-C32-H32B	109.5	H2AC-C2A-H2AB	110.1
H32A-C32-H32B	109.5	H2AA-C2A-H2AB	107.9
C14-C32-H32C	109.7	C9B-N1B-C5B	111.6(3)
H32A-C32-H32C	109.5	C9B-N1B-C1B	115.0(4)
H32B-C32-H32C	109.5	C5B-N1B-C1B	107.8(4)
C19-C33-H33A	109.7	C9B-N1B-C13B	84.4(4)
C19-C33-H33B	110.0	C5B-N1B-C13B	121.0(5)
H33A-C33-H33B	109.5	C1B-N1B-C13B	115.5(4)
C19-C33-H33C	108.7	C9B-N1B-C13C	121.5(5)
H33A-C33-H33C	109.5	C5B-N1B-C13C	95.0(4)
H33B-C33-H33C	109.5	C1B-N1B-C13C	103.5(5)
C19-C34-H34A	109.7	C2B-C1B-N1B	118.6(4)
C19-C34-H34B	109.4	C2B-C1B-H1BA	107.5
H34A-C34-H34B	109.5	N1B-C1B-H1BA	108.9
C19-C34-H34C	109.4	C2B-C1B-H1BB	107.5
H34A-C34-H34C	109.5	N1B-C1B-H1BB	106.3
H34B-C34-H34C	109.5	H1BA-C1B-H1BB	107.5
C28-C35-H35A	110.2	C1B-C2B-C3B	109.1(5)
C28-C35-H35B	108.5	C1B-C2B-H2BA	109.5

C3B-C2B-H2BA	109.9	H8BA-C8B-H8BB	109.5
C1B-C2B-H2BB	109.4	C7B-C8B-H8BC	108.6
C3B-C2B-H2BB	110.3	H8BA-C8B-H8BC	109.5
H2BA-C2B-H2BB	108.7	H8BB-C8B-H8BC	109.5
C4B-C3B-C2B	110.3(4)	C10B-C9B-N1B	113.3(4)
C4B-C3B-H3BA	109.4	C10B-C9B-H9BA	108.1
C2B-C3B-H3BA	109.4	N1B-C9B-H9BA	109.7
C4B-C3B-H3BB	109.8	C10B-C9B-H9BB	108.8
C2B-C3B-H3BB	109.6	N1B-C9B-H9BB	108.6
H3BA-C3B-H3BB	108.3	H9BA-C9B-H9BB	108.2
C3B-C4B-H4BA	110.0	C9B-C10B-C11B	113.1(4)
C3B-C4B-H4BB	109.4	C9B-C10B-H10A	107.6
H4BA-C4B-H4BB	109.5	C11B-C10B-H10A	110.6
C3B-C4B-H4BC	109.0	C9B-C10B-H10B	108.9
H4BA-C4B-H4BC	109.5	C11B-C10B-H10B	108.5
H4BB-C4B-H4BC	109.5	H10A-C10B-H10B	108.0
C6B-C5B-N1B	116.7(3)	C12B-C11B-C10B	115.9(5)
C6B-C5B-H5BA	108.1	C12B-C11B-H11A	108.4
N1B-C5B-H5BA	108.4	C10B-C11B-H11A	108.4
C6B-C5B-H5BB	107.3	C12B-C11B-H11B	106.9
N1B-C5B-H5BB	108.2	C10B-C11B-H11B	109.0
H5BA-C5B-H5BB	107.8	H11A-C11B-H11B	108.0
C5B-C6B-C7B	110.8(3)	C11B-C12B-H12A	112.7
C5B-C6B-H6BA	109.7	C11B-C12B-H12B	107.1
C7B-C6B-H6BA	109.5	H12A-C12B-H12B	109.5
C5B-C6B-H6BB	108.3	C11B-C12B-H12C	108.6
C7B-C6B-H6BB	109.9	H12A-C12B-H12C	109.5
H6BA-C6B-H6BB	108.5	H12B-C12B-H12C	109.5
C6B-C7B-C8B	113.7(4)	C14B-C13B-N1B	108.3(5)
C6B-C7B-H7BA	108.7	C14B-C13B-H13A	107.3
C8B-C7B-H7BA	108.5	N1B-C13B-H13A	109.1
C6B-C7B-H7BB	109.0	C14B-C13B-H13B	110.9
C8B-C7B-H7BB	108.7	N1B-C13B-H13B	112.3
H7BA-C7B-H7BB	108.0	H13A-C13B-H13B	108.9
C7B-C8B-H8BA	111.0	H13D-C13B-H14C	116.2
C7B-C8B-H8BB	108.8	C13B-C14B-C15B	111.0(6)

C13B-C14B-H14A	106.7	H14C-C14C-H14D	109.1
C15B-C14B-H14A	108.6	C16B-C15B-C14B	129.6(5)
C13B-C14B-H14B	109.9	C16B-C15B-H15A	104.8
C15B-C14B-H14B	112.0	C14B-C15B-H15A	104.4
H14A-C14B-H14B	108.4	C16B-C15B-H15B	104.6
C15B-C14B-H13D	111.2	C14C-C15B-H15B	107.8
H13C-C14B-H14D	113.1	C14B-C15B-H15B	105.5
C14C-C13C-N1B	111.8(6)	H15A-C15B-H15B	106.1
C14C-C13C-H13C	110.1	C15B-C16B-H16C	111.5
N1B-C13C-H13C	108.9	C15B-C16B-H16A	109.3
C14C-C13C-H13D	109.0	H16C-C16B-H16A	110.1
N1B-C13C-H13D	108.4	C15B-C16B-H16B	107.8
H13C-C13C-H13D	108.5	H16C-C16B-H16B	110.1
C13C-C14C-C15B	104.3(6)	H16A-C16B-H16B	108.0
C13C-C14C-H14C	110.8	C12D-C1C-C11D	121.2(8)
C15B-C14C-H14C	111.7	C11E-C1C-C12E	110.7(5)
C13C-C14C-H14D	110.0	C12C-C1C-C11C	110.2(3)
C15B-C14C-H14D	110.9		

Table 4. Anisotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for $7 \cdot (\text{C}_4\text{H}_9)_4\text{NCH}_3\text{CO}_2 \cdot \text{CH}_2\text{Cl}_2$. The anisotropic displacement factor exponent takes the form: $-2\pi^2 [h^2 a^{*2} U^{11} + \dots + 2 h k a^* b^* U^{12}]$.

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
S1	63(1)	51(1)	45(1)	-12(1)	15(1)	-22(1)
S2	41(1)	54(1)	40(1)	-20(1)	3(1)	-17(1)
N1	25(2)	44(2)	45(2)	-21(2)	2(2)	-9(2)
N2	38(2)	31(2)	41(2)	-13(2)	6(2)	-11(2)
N3	37(2)	35(2)	33(2)	-15(1)	8(1)	-7(1)
N4	47(2)	30(2)	47(2)	-18(2)	8(1)	-16(2)
C1	48(2)	41(2)	49(2)	-14(2)	14(2)	-25(2)
C2	48(2)	54(2)	52(2)	-26(2)	10(2)	-18(2)
C3	42(2)	55(2)	52(2)	-21(2)	2(2)	-17(2)
C4	37(2)	43(2)	49(2)	-19(2)	13(2)	-25(2)
C5	39(2)	49(2)	50(2)	-25(2)	15(2)	-21(2)
C6	28(2)	50(2)	49(2)	-30(2)	11(2)	-17(2)
C7	29(2)	57(2)	61(2)	-36(2)	9(2)	-12(2)
C8	37(2)	46(2)	49(2)	-26(2)	-1(2)	-2(2)
C9	31(2)	39(2)	42(2)	-22(2)	2(2)	-5(2)
C10	36(2)	32(2)	39(2)	-19(2)	2(2)	-4(2)
C11	48(2)	34(2)	47(2)	-17(2)	-5(2)	3(2)
C12	65(3)	34(2)	46(2)	-11(2)	11(2)	-16(2)
C13	50(2)	32(2)	42(2)	-19(2)	15(2)	-13(2)
C14	48(2)	41(2)	51(2)	-25(2)	18(2)	-22(2)
C15	32(2)	39(2)	40(2)	-19(2)	8(2)	-15(2)
C16	38(2)	39(2)	45(2)	-17(2)	-7(2)	0(2)
C17	44(2)	41(2)	38(2)	-21(2)	3(2)	-7(2)
C18	29(2)	34(2)	34(2)	-11(2)	6(2)	-11(2)
C19	29(2)	40(2)	41(2)	-8(2)	6(2)	-11(2)
C20	26(2)	36(2)	32(2)	-6(2)	5(2)	0(2)
C21	36(2)	44(2)	38(2)	-10(2)	11(2)	3(2)
C22	40(2)	39(2)	40(2)	-18(2)	4(2)	6(2)
C23	33(2)	29(2)	33(2)	-12(2)	-2(2)	1(2)

C24	36(2)	34(2)	39(2)	-16(2)	2(2)	-7(2)
C25	54(2)	38(2)	61(2)	-30(2)	3(2)	-8(2)
C26	63(3)	34(2)	75(3)	-23(2)	1(2)	-19(2)
C27	43(2)	31(2)	54(2)	-11(2)	4(2)	-14(2)
C28	60(3)	40(2)	58(2)	-8(2)	15(2)	-28(2)
C29	59(2)	62(2)	51(2)	-29(2)	14(2)	-27(2)
C30	50(2)	79(3)	66(2)	-37(2)	28(2)	-37(2)
C31	57(2)	83(3)	70(3)	-52(2)	18(2)	-34(2)
C32	79(3)	44(2)	102(3)	-39(2)	45(2)	-35(2)
C33	38(2)	52(2)	63(2)	-4(2)	-13(2)	-14(2)
C34	38(2)	53(2)	70(3)	-8(2)	21(2)	-11(2)
C35	67(3)	55(2)	51(2)	-3(2)	11(2)	-15(2)
C36	83(3)	44(2)	102(3)	-12(2)	27(3)	-36(2)
O1A	37(1)	41(1)	47(1)	-19(1)	6(1)	-9(1)
O2A	39(1)	40(1)	51(1)	-22(1)	9(1)	-11(1)
C1A	39(2)	47(2)	40(2)	-17(2)	10(2)	-22(2)
C2A	53(2)	56(2)	45(2)	-25(2)	4(2)	-17(2)
N1B	68(3)	117(3)	77(3)	-32(3)	4(2)	-16(3)
C1B	70(4)	93(4)	93(4)	-15(3)	11(3)	6(3)
C2B	69(4)	142(5)	128(5)	15(4)	1(3)	1(4)
C3B	96(4)	72(3)	114(4)	-33(3)	-9(3)	9(3)
C4B	124(5)	112(5)	148(5)	-35(4)	-3(4)	-28(4)
C5B	65(3)	93(3)	92(3)	-61(3)	12(3)	-11(3)
C6B	85(3)	79(3)	79(3)	-38(3)	0(3)	-27(3)
C7B	65(3)	103(4)	100(4)	-62(3)	0(3)	-12(3)
C8B	118(5)	133(5)	131(5)	-24(4)	-7(4)	-66(4)
C9B	152(6)	167(6)	90(4)	12(4)	-46(4)	-97(5)
C10B	137(5)	71(3)	126(4)	-30(3)	-69(4)	-17(3)
C11B	199(6)	95(4)	66(3)	-29(3)	9(3)	-78(4)
C12B	291(8)	115(5)	81(4)	-5(3)	-48(4)	-119(5)
C13B	79(7)	144(9)	76(6)	-72(6)	13(5)	-42(7)
C14B	115(9)	75(7)	102(7)	-63(6)	-15(7)	-3(7)
C13C	76(10)	42(7)	54(6)	-30(6)	-17(7)	12(6)
C14C	46(7)	61(7)	78(8)	-31(6)	20(6)	-13(6)
C15B	94(3)	69(3)	51(2)	-16(2)	-8(2)	-18(3)
C16B	181(6)	164(5)	84(3)	-60(3)	35(3)	-127(5)

Cl1C	139(2)	122(2)	69(1)	-26(1)	-22(1)	-57(2)
Cl2C	91(2)	162(2)	75(1)	-49(1)	2(1)	-67(2)
C1C	169(5)	69(3)	81(3)	-13(3)	-30(3)	-24(3)
Cl1D	137(10)	183(10)	150(10)	-77(8)	25(8)	-50(8)
Cl1E	325(12)	127(6)	171(8)	-97(6)	-64(8)	11(7)
Cl2E	276(12)	251(11)	274(11)	-114(8)	-52(8)	-84(8)
Cl2D	201(13)	220(13)	226(12)	-88(10)	35(9)	-34(9)

Table 5. Hydrogen coordinates ($\times 10^4$) and isotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for $7 \cdot (\text{C}_4\text{H}_9)_4\text{NCH}_3\text{CO}_2 \cdot \text{CH}_2\text{Cl}_2$.

	x	y	z	U(eq)
H2	10558	3711	8029	59
H3	12024	2094	7993	58
H7	14394	21	7565	54
H8	13923	-1410	8669	53
H11	12436	-2497	9599	55
H12	10547	-2972	9852	59
H16	8690	-748	10032	51
H17	7219	811	10142	49
H21	4703	3294	9996	53
H22	5724	4677	9631	52
H25	7231	5731	8867	59
H26	8503	6334	7760	66
H29A	10943	1385	6017	80
H29B	11489	2136	5389	80
H29C	12151	948	5707	80
H30A	13858	1946	6666	87
H30B	13925	1294	6103	87
H30C	13263	2482	5785	87
H31A	8357	-687	7543	91
H31B	8626	-1873	7723	91
H31C	7359	-1193	7782	91
H32A	8407	-2590	9836	102
H32B	7388	-2363	9195	102
H32C	8655	-3043	9136	102
H33A	5127	2509	7768	82
H33B	4350	1788	8086	82
H33C	3941	2910	8117	82
H34A	4765	1384	10161	87
H34B	3720	2233	9569	87
H34C	4129	1111	9538	87

H35A	7840	4728	6101	94
H35B	7576	5922	5865	94
H35C	8589	5288	5493	94
H36A	10132	5914	6849	114
H36B	9991	6023	5942	114
H36C	8977	6658	6314	114
H2AC	10093	1050	8679	74
H2AA	8854	1394	8991	74
H2AB	9476	2210	8595	74
H1N	11030(20)	560(20)	7466(15)	26(9)
H2N	9870(30)	-340(20)	8132(17)	46(11)
H3N	6760(20)	2950(20)	8315(16)	37(10)
H4N	7810(20)	3945(19)	7586(14)	15(8)
H1BA	6084	4292	1699	117
H1BB	6935	3249	1696	117
H2BA	8479	3629	2101	164
H2BB	7636	4655	2162	164
H3BA	7160	5341	744	122
H3BB	7828	4284	655	122
H4BA	8918	5375	267	198
H4BB	8875	5493	1117	198
H4BC	9548	4428	1027	198
H5BA	5766	2317	2445	95
H5BB	4877	3358	2425	95
H6BA	4750	2744	3765	94
H6BB	5746	1729	3837	94
H7BA	4553	1317	3070	102
H7BB	3564	2338	2979	102
H8BA	3154	974	3945	189
H8BB	4353	715	4428	189
H8BC	3355	1745	4336	189
H9BA	6954	4004	3426	167
H9BB	6122	3399	3863	167
H10A	4652	4554	2899	137
H10B	5493	5195	2568	137
H11A	4679	4910	4137	134

H11B	4293	5958	3437	134
H12A	5622	6015	4344	233
H12B	6518	5048	4216	233
H12C	6129	6107	3510	233
H13A	7369	2183	3938	107
H13B	8107	2892	3500	107
H14A	8579	1984	2642	113
H14B	7791	1308	3034	113
H13C	7701	1856	2606	73
H13D	7229	1557	3449	73
H14C	8422	2325	3880	73
H14D	8967	2477	3056	73
H15A	9747	417	3499	89
H15B	9041	557	4206	89
H16C	11025	558	4230	182
H16A	10084	1340	4539	182
H16B	10473	1716	3680	182

Table 6. Torsion angles [°] for **7**·(C₄H₉)₄NCH₃CO₂·CH₂Cl₂.

C4-S1-C1-C2	0.6(3)	C8-C9-C10-N2	-169.7(3)
C4-S1-C1-C28	-179.7(2)	N1-C9-C10-N2	5.0(4)
C28-C1-C2-C3	179.5(3)	N2-C10-C11-C12	-0.1(3)
S1-C1-C2-C3	-0.8(4)	C9-C10-C11-C12	-175.1(3)
C1-C2-C3-C4	0.6(4)	C10-C11-C12-C13	0.1(4)
C2-C3-C4-C5	-178.9(3)	C11-C12-C13-N2	0.0(3)
C2-C3-C4-S1	-0.1(3)	C11-C12-C13-C14	-178.9(3)
C1-S1-C4-C3	-0.3(2)	C10-N2-C13-C12	0.0(3)
C1-S1-C4-C5	178.6(2)	C10-N2-C13-C14	179.0(2)
C3-C4-C5-C6	30.0(4)	C12-C13-C14-C15	109.2(4)
S1-C4-C5-C6	-148.6(2)	N2-C13-C14-C15	-69.6(3)
C3-C4-C5-C30	-90.5(4)	C12-C13-C14-C32	-9.8(5)
S1-C4-C5-C30	90.9(3)	N2-C13-C14-C32	171.4(2)
C3-C4-C5-C29	150.0(3)	C12-C13-C14-C31	-129.3(4)
S1-C4-C5-C29	-28.6(3)	N2-C13-C14-C31	52.0(3)
C9-N1-C6-C7	0.2(3)	C13-C14-C15-C16	-40.9(4)
C9-N1-C6-C5	179.8(3)	C32-C14-C15-C16	78.5(4)
C4-C5-C6-C7	-118.9(4)	C31-C14-C15-C16	-161.6(3)
C30-C5-C6-C7	1.6(5)	C13-C14-C15-S2	140.9(2)
C29-C5-C6-C7	121.1(4)	C32-C14-C15-S2	-99.7(3)
C4-C5-C6-N1	61.7(3)	C31-C14-C15-S2	20.2(3)
C30-C5-C6-N1	-177.9(2)	C18-S2-C15-C16	0.8(2)
C29-C5-C6-N1	-58.4(3)	C18-S2-C15-C14	179.3(2)
N1-C6-C7-C8	0.0(3)	C14-C15-C16-C17	-178.9(3)
C5-C6-C7-C8	-179.5(3)	S2-C15-C16-C17	-0.5(3)
C6-C7-C8-C9	-0.3(3)	C15-C16-C17-C18	-0.2(4)
C7-C8-C9-N1	0.4(3)	C16-C17-C18-C19	177.9(3)
C7-C8-C9-C10	175.7(3)	C16-C17-C18-S2	0.8(3)
C6-N1-C9-C8	-0.4(3)	C15-S2-C18-C17	-0.9(2)
C6-N1-C9-C10	-176.4(2)	C15-S2-C18-C19	-178.2(2)
C13-N2-C10-C11	0.1(3)	C17-C18-C19-C20	48.2(4)
C13-N2-C10-C9	175.7(2)	S2-C18-C19-C20	-135.0(2)
C8-C9-C10-C11	4.6(5)	C17-C18-C19-C33	168.2(3)
N1-C9-C10-C11	179.4(3)	S2-C18-C19-C33	-14.9(3)

C17-C18-C19-C34	-72.9(4)	C2-C1-C28-C27	-31.6(4)
S2-C18-C19-C34	103.9(3)	S1-C1-C28-C27	148.8(2)
C23-N3-C20-C21	0.4(3)	C2-C1-C28-C35	-152.8(3)
C23-N3-C20-C19	175.5(3)	S1-C1-C28-C35	27.5(4)
C18-C19-C20-N3	58.8(3)	C2-C1-C28-C36	88.2(4)
C33-C19-C20-N3	-62.6(4)	S1-C1-C28-C36	-91.4(3)
C34-C19-C20-N3	179.3(3)	C9B-N1B-C1B-C2B	57.3(6)
C18-C19-C20-C21	-127.6(3)	C5B-N1B-C1B-C2B	-177.5(4)
C33-C19-C20-C21	111.0(4)	C13B-N1B-C1B-C2B	-38.6(6)
C34-C19-C20-C21	-7.1(5)	C13C-N1B-C1B-C2B	-77.6(6)
N3-C20-C21-C22	-0.1(3)	N1B-C1B-C2B-C3B	-178.4(4)
C19-C20-C21-C22	-174.5(3)	C1B-C2B-C3B-C4B	-171.6(4)
C20-C21-C22-C23	-0.2(3)	C9B-N1B-C5B-C6B	-49.9(5)
C21-C22-C23-N3	0.5(3)	C1B-N1B-C5B-C6B	-177.2(3)
C21-C22-C23-C24	177.0(3)	C13B-N1B-C5B-C6B	46.7(6)
C20-N3-C23-C22	-0.6(3)	C13C-N1B-C5B-C6B	76.9(6)
C20-N3-C23-C24	-177.4(3)	N1B-C5B-C6B-C7B	-173.8(3)
C27-N4-C24-C25	-0.4(3)	C5B-C6B-C7B-C8B	-179.8(4)
C27-N4-C24-C23	176.4(3)	C5B-N1B-C9B-C10B	-64.5(5)
C22-C23-C24-C25	4.1(5)	C1B-N1B-C9B-C10B	58.7(6)
N3-C23-C24-C25	-179.9(3)	C13B-N1B-C9B-C10B	174.3(5)
C22-C23-C24-N4	-171.9(3)	C13C-N1B-C9B-C10B	-175.2(6)
N3-C23-C24-N4	4.1(4)	N1B-C9B-C10B-C11B	172.8(4)
N4-C24-C25-C26	0.6(4)	C9B-C10B-C11B-C12B	58.2(6)
C23-C24-C25-C26	-175.9(3)	C9B-N1B-C13B-C14B	-178.2(7)
C24-C25-C26-C27	-0.5(4)	C5B-N1B-C13B-C14B	69.9(8)
C25-C26-C27-N4	0.3(4)	C1B-N1B-C13B-C14B	-63.2(8)
C25-C26-C27-C28	178.0(3)	C13C-N1B-C13B-C14B	16.2(10)
C24-N4-C27-C26	0.1(4)	N1B-C13B-C14B-C15B	-176.8(6)
C24-N4-C27-C28	-178.0(3)	C9B-N1B-C13C-C14C	-42.9(12)
C26-C27-C28-C1	120.2(4)	C5B-N1B-C13C-C14C	-162.1(9)
N4-C27-C28-C1	-62.3(4)	C1B-N1B-C13C-C14C	88.2(10)
C26-C27-C28-C35	-117.8(4)	N1B-C13C-C14C-C15B	172.9(7)
N4-C27-C28-C35	59.6(4)	C13C-C14C-C15B-C16B	-179.7(8)
C26-C27-C28-C36	1.2(5)	C13C-C14C-C15B-C14B	19.5(10)
N4-C27-C28-C36	178.7(3)	C13B-C14B-C15B-C16B	-55.1(11)

Table 7. Hydrogen bonds for **7**·(C₄H₉)₄NCH₃CO₂·CH₂Cl₂ [Å and °].

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)
N1-H1N...O1A	0.81(3)	2.06(3)	2.864(4)	168(3)
N2-H2N...O1A	0.84(3)	2.03(3)	2.855(4)	169(3)
N3-H3N...O2A	0.82(3)	2.04(3)	2.856(3)	169(3)
N4-H4N...O2A	0.82(2)	2.07(2)	2.869(3)	167(2)

Figure 1. View of the macrocycle acetate complex in $7 \cdot (\text{C}_4\text{H}_9)_4\text{NCH}_3\text{CO}_2 \cdot \text{CH}_2\text{Cl}_2$ showing the atom labeling scheme. Displacement ellipsoids are scaled to the 50% probability level. The methyl hydrogen atoms have been removed for clarity. Dashed lines are indicative of H-bonding interactions, which are listed in Table 7.

Figure 2. View of the tetrakis-n-butyl ammonium ion in $7 \cdot (\text{C}_4\text{H}_9)_4\text{NCH}_3\text{CO}_2 \cdot \text{CH}_2\text{Cl}_2$ showing the atom labeling scheme. Displacement ellipsoids are scaled to the 30% probability level. The hydrogen atoms have been removed for clarity. A portion of the cation was found to be disordered. C13b and C14b refined to a site occupancy of 59(2)% while C13c and C14c refined to a site occupancy factor of 41(2)%.

Figure 3. Unit cell packing diagram for $7 \cdot (\text{C}_4\text{H}_9)_4\text{NCH}_3\text{CO}_2 \cdot \text{CH}_2\text{Cl}_2$.

Crystallographic Material for $7 \cdot (\text{C}_4\text{H}_9)_4\text{NCl} \cdot \text{H}_2\text{O} \cdot 2\text{CH}_2\text{Cl}_2$.

- X-ray Experimental.
- Table 1. Crystallographic Data for $7 \cdot (\text{C}_4\text{H}_9)_4\text{NCl} \cdot \text{H}_2\text{O} \cdot 2\text{CH}_2\text{Cl}_2$.
- Table 2. Fractional coordinates and equivalent isotropic thermal parameters ($\text{\AA}^2$) for the non-hydrogen atoms of $7 \cdot (\text{C}_4\text{H}_9)_4\text{NCl} \cdot \text{H}_2\text{O} \cdot 2\text{CH}_2\text{Cl}_2$.
- Table 3. Bond Lengths ($\text{\AA}$) and Angles ($^\circ$) for the non-hydrogen atoms of $7 \cdot (\text{C}_4\text{H}_9)_4\text{NCl} \cdot \text{H}_2\text{O} \cdot 2\text{CH}_2\text{Cl}_2$.
- Table 4. Anisotropic thermal parameters for the non-hydrogen atoms of $7 \cdot (\text{C}_4\text{H}_9)_4\text{NCl} \cdot \text{H}_2\text{O} \cdot 2\text{CH}_2\text{Cl}_2$.
- Table 5. Fractional coordinates and isotropic thermal parameters ($\text{\AA}^2$) for the hydrogen atoms of $7 \cdot (\text{C}_4\text{H}_9)_4\text{NCl} \cdot \text{H}_2\text{O} \cdot 2\text{CH}_2\text{Cl}_2$.
- Table 6. Torsion Angles ($^\circ$) for the non-hydrogen atoms of $7 \cdot (\text{C}_4\text{H}_9)_4\text{NCl} \cdot \text{H}_2\text{O} \cdot 2\text{CH}_2\text{Cl}_2$.
- Table 7. Hydrogen Bond Lengths ($\text{\AA}$) and Angles ($^\circ$) for $7 \cdot (\text{C}_4\text{H}_9)_4\text{NCl} \cdot \text{H}_2\text{O} \cdot 2\text{CH}_2\text{Cl}_2$.
- Figure 1. View of the macrocycle in $7 \cdot (\text{C}_4\text{H}_9)_4\text{NCl} \cdot \text{H}_2\text{O} \cdot 2\text{CH}_2\text{Cl}_2$ showing the disorder of one of the thiophene moieties.
- Figure 2. View of the cation in $7 \cdot (\text{C}_4\text{H}_9)_4\text{NCl} \cdot \text{H}_2\text{O} \cdot 2\text{CH}_2\text{Cl}_2$ showing the atom labeling scheme.

X-ray Experimental for $7 \cdot (\text{C}_4\text{H}_9)_4\text{NCl} \cdot \text{H}_2\text{O} \cdot 2\text{CH}_2\text{Cl}_2$: $\text{C}_{54}\text{H}_{82}\text{Cl}_5\text{N}_5\text{OS}_2$, crystals grew as colorless plates by vapor diffusion of pentane into a methylene chloride solution of the macrocycle complex. The crystals grew cloudy very quickly when removed from the mother liquor. The crystals were stabilized only very briefly by immersion in mineral oil. The data crystal was cut from a larger crystal and had approximate dimensions; $0.36 \times 0.28 \times 0.15$ mm. The data were collected on a Nonius Kappa CCD diffractometer using a graphite monochromator with $\text{MoK}\alpha$ radiation ($\lambda = 0.71073 \text{ \AA}$). A total of 365 frames of data were collected using ω -scans with a scan range of 0.8° and a counting time of 145 seconds per frame. The data were collected at 153 K using an Oxford Cryostream low temperature device. Details of crystal data, data collection and structure refinement are listed in Table 1. Data reduction were performed using DENZO-SMN.¹ The structure was solved by direct methods using SIR97² and refined by full-matrix least-squares on F^2 with anisotropic displacement parameters for the non-H atoms using SHELXL-97.³ The hydrogen atoms on carbon and nitrogen were calculated in ideal positions with isotropic displacement parameters set to $1.2x\text{Ueq}$ of the attached atom ($1.5x\text{Ueq}$ for methyl hydrogen atoms). The hydrogen atoms on the water molecule were observed in a ΔF map and refined with isotropic displacement parameters.

One of the thiophene rings of the macrocycle was disordered by rotation about the meso carbons to which the ring is attached to the macrocycle (Figure 2). The disorder was modeled by assigning the site occupancy of the atoms of one thiophene ring to a variable x and the other to $(1-x)$. While refining the site occupancies of the two rings, a common isotropic displacement parameter was refined for the eight carbon atoms and a second isotropic displacement parameter for the two S atoms. In this way, the site occupancy for the minor component refined to 21(2)% for atoms S2a, C24a, C25a, C26a and C27a. Finally, these atoms of the major component of the

disordered ring were refined anisotropically while restraining their displacement parameters to be approximately isotropic. One CH₃-CH₂- moiety of the cation was also disordered. The modeling of the disorder was done in the same fashion. The site occupancy factor for the major component (C3a and C4a) refined to 60(2)%.

There appeared to be two molecules of methylene chloride in the crystal lattice and near the cation. The molecules resided in a columnar void. These solvate molecules appeared to be badly disordered. Attempts at modeling the atoms failed. The contribution to the scattering by this solvent region was removed by use of the utility SQUEEZE in Platon.⁴

The function, $\sum w(|F_o|^2 - |F_c|^2)^2$, was minimized, where $w = 1/[(\sigma(F_o))^2 + (0.0 \cdot P)^2]$ and $P = (|F_o|^2 + 2|F_c|^2)/3$. $R_w(F^2)$ refined to 0.158, with $R(F)$ equal to 0.0956 and a goodness of fit, S , = 1.78. Definitions used for calculating $R(F)$, $R_w(F^2)$ and the goodness of fit, S , are given below.⁵ The data were corrected for secondary extinction effects. The correction takes the form: $F_{corr} = kF_c/[1 + (1.2(2) \times 10^{-6}) \cdot F_c^2 \lambda^3/(\sin 2\theta)]^{0.25}$ where k is the overall scale factor. Neutral atom scattering factors and values used to calculate the linear absorption coefficient are from the International Tables for X-ray Crystallography (1992).⁶ All figures were generated using SHELXTL/PC.⁷ Tables of positional and thermal parameters, bond lengths and angles, torsion angles, H-bonding interactions, figures and lists of observed and calculated structure factors are located in tables 1 through 8.

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6. $R_w(F^2) = \{ \sum w(|F_o|^2 - |F_c|^2)^2 / \sum w(|F_o|^4) \}^{1/2}$ where w is the weight given each reflection.
 $R(F) = \sum (|F_o| - |F_c|) / \sum |F_o|$ for reflections with $F_o > 4(\sigma(F_o))$.
 $S = [\sum w(|F_o|^2 - |F_c|^2)^2 / (n - p)]^{1/2}$, where n is the number of reflections and p is the number of refined parameters.
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Table 1. Crystal data and structure refinement for $7 \cdot (\text{C}_4\text{H}_9)_4\text{NCl} \cdot \text{H}_2\text{O} \cdot 2\text{CH}_2\text{Cl}_2$.

Empirical formula	C ₅₄ H ₈₂ Cl ₅ N ₅ O ₂	
Formula weight	1058.62	
Temperature	153(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	$P2_1/n$	
Unit cell dimensions	$a = 15.1902(5)$ Å	$\alpha = 90^\circ$.
	$b = 18.6941(6)$ Å	$\beta = 98.979(2)^\circ$.
	$c = 20.9059(8)$ Å	$\gamma = 90^\circ$.
Volume	5863.8(4) Å ³	
Z	4	
Density (calculated)	1.199 g cm ⁻³	
Absorption coefficient	0.359 mm ⁻¹	
F(000)	2264	
Crystal size	0.36 × 0.28 × 0.15 mm	
Theta range for data collection	2.93 to 25.00°.	
Index ranges	-18 ≤ h ≤ 18, -22 ≤ k ≤ 20, -24 ≤ l ≤ 24	
Reflections collected	17723	
Independent reflections	10250 [R(int) = 0.0718]	
Completeness to theta = 25.00°	99.3 %	
Absorption correction	None	
Refinement method	Full-matrix least-squares on F ²	
Data / restraints / parameters	10250 / 379 / 603	
Goodness-of-fit on F ²	1.727	
Final R indices [I > 2σ(I)]	R1 = 0.0956, wR2 = 0.1495	
R indices (all data)	R1 = 0.1892, wR2 = 0.1583	
Extinction coefficient	$1.2(2) \times 10^{-6}$	
Largest diff. peak and hole	0.384 and -0.256 e.Å ⁻³	

Table 2. Atomic coordinates ($\times 10^4$) and equivalent isotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for $7 \cdot (\text{C}_4\text{H}_9)_4\text{NCl} \cdot \text{H}_2\text{O} \cdot 2\text{CH}_2\text{Cl}_2$. U(eq) is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U(eq)
S2	2205(1)	2794(1)	171(1)	45(1)
C24	2710(3)	2806(4)	-496(2)	38(2)
C25	2739(4)	3499(3)	-712(3)	58(2)
C26	2334(4)	3985(3)	-338(3)	52(2)
C27	2000(6)	3669(3)	179(3)	45(4)
S2A	2786(4)	3217(4)	112(3)	47(2)
C27A	1848(10)	3655(7)	116(7)	120(40)
C26A	1123(18)	3040(12)	-202(11)	75(9)
C25A	1521(11)	2557(12)	-589(11)	84(9)
C24A	2441(10)	2594(6)	-438(5)	93(18)
S1	7232(1)	4041(1)	2117(1)	41(1)
Cl1	5126(1)	3782(1)	-242(1)	48(1)
O1W	4180(3)	4791(3)	762(2)	43(1)
N1	2875(2)	4224(2)	1532(2)	34(1)
N2	4670(2)	4642(2)	2218(2)	34(1)
N3	6370(2)	2577(2)	630(2)	33(1)
N4	4545(2)	2153(2)	-69(2)	37(1)
C1	2027(3)	3947(2)	1340(2)	35(1)
C2	1800(3)	3618(2)	1881(3)	46(1)
C3	2502(3)	3697(2)	2388(2)	41(1)
C4	3165(3)	4071(2)	2172(2)	33(1)
C5	4032(3)	4303(2)	2514(2)	37(1)
C6	4360(3)	4246(3)	3165(2)	52(2)
C7	5236(3)	4562(3)	3257(3)	54(2)
C8	5406(3)	4795(2)	2665(2)	36(1)
C9	6195(3)	5185(2)	2485(2)	38(1)
C10	6550(3)	4791(2)	1957(2)	35(1)
C11	6410(3)	4902(2)	1304(2)	39(1)

C12	6815(3)	4407(2)	940(2)	38(1)
C13	7296(3)	3900(2)	1310(2)	37(1)
C14	7778(3)	3255(3)	1092(2)	41(1)
C15	7165(3)	2606(3)	1038(2)	37(1)
C16	7259(3)	1968(3)	1376(2)	48(1)
C17	6516(4)	1554(3)	1138(2)	52(2)
C18	5952(3)	1935(2)	685(2)	37(1)
C19	5125(3)	1707(2)	315(2)	34(1)
C20	4742(3)	1051(3)	286(2)	47(1)
C21	3891(3)	1099(3)	-109(2)	51(1)
C22	3781(3)	1791(3)	-325(2)	40(1)
C23	3044(3)	2132(3)	-792(2)	43(1)
C28	1535(3)	4054(2)	670(2)	36(1)
C29	5906(3)	5935(2)	2255(2)	46(1)
C30	6916(3)	5252(2)	3095(2)	52(2)
C31	8632(3)	3104(3)	1571(2)	52(2)
C32	8042(3)	3401(3)	419(2)	58(2)
C33	3432(4)	2379(3)	-1418(3)	85(2)
C34	2318(4)	1596(3)	-1006(3)	84(2)
C35	1500(4)	4849(3)	503(3)	72(2)
C36	572(3)	3787(3)	640(3)	81(2)
N1A	2705(3)	1224(2)	1972(2)	48(1)
C1A	1931(4)	922(3)	1518(3)	65(2)
C2A	1203(4)	1435(3)	1268(3)	85(2)
C3A	639(9)	1109(8)	654(7)	138(8)
C4A	-167(7)	1480(6)	496(6)	121(5)
C3AA	388(8)	1088(8)	888(7)	71(6)
C4AA	517(13)	845(12)	273(8)	140(9)
C5A	3325(4)	615(3)	2211(3)	57(2)
C6A	3788(4)	259(3)	1713(3)	84(2)
C7A	4422(5)	-302(3)	2013(3)	99(2)
C8A	4961(5)	-691(4)	1579(4)	136(3)
C9A	3188(3)	1784(2)	1626(2)	42(1)
C10A	4031(3)	2074(3)	1969(2)	50(1)
C11A	4381(3)	2659(2)	1589(2)	46(1)
C12A	5271(3)	2960(3)	1907(3)	68(2)

C13A	2385(3)	1589(3)	2547(3)	53(2)
C14A	1809(4)	1147(3)	2926(3)	68(2)
C15A	1592(4)	1575(4)	3498(3)	84(2)
C16A	1013(4)	2232(3)	3308(3)	91(2)

Table 3. Bond lengths [Å] and angles [°] for $7 \cdot (\text{C}_4\text{H}_9)_4\text{NCl} \cdot \text{H}_2\text{O} \cdot 2\text{CH}_2\text{Cl}_2$.

S2-C27	1.665(5)	C1-C2	1.379(6)
S2-C24	1.694(6)	C1-C28	1.493(6)
C24-C25	1.375(7)	C2-C3	1.389(6)
C24-C23	1.524(6)	C2-H2A	0.96
C25-C26	1.403(8)	C3-C4	1.360(6)
C25-H25	0.96	C3-H3A	0.96
C26-C27	1.395(7)	C4-C5	1.462(6)
C26-H26	0.96	C5-C6	1.379(6)
C27-C28	1.516(6)	C6-C7	1.441(6)
S2A-C27A	1.645(9)	C6-H6A	0.96
S2A-C24A	1.663(9)	C7-C8	1.375(6)
C27A-C28	1.516(10)	C7-H7A	0.96
C27A-C26A	1.66(3)	C8-C9	1.500(6)
C26A-C25A	1.410(13)	C9-C10	1.496(6)
C26A-H26A	0.96	C9-C29	1.525(6)
C25A-C24A	1.386(10)	C9-C30	1.550(6)
C25A-H25A	0.96	C10-C11	1.364(6)
C24A-C23	1.531(9)	C11-C12	1.400(6)
S1-C13	1.725(5)	C11-H11A	0.96
S1-C10	1.743(4)	C12-C13	1.363(6)
O1W-H1WA	0.83(6)	C12-H12A	0.96
O1W-H1WB	0.70(8)	C13-C14	1.518(6)
N1-C4	1.373(5)	C14-C15	1.522(6)
N1-C1	1.389(5)	C14-C31	1.536(6)
N1-H1N	0.90	C14-C32	1.546(6)
N2-C8	1.370(5)	C15-C16	1.382(6)
N2-C5	1.381(5)	C16-C17	1.395(6)
N2-H2N	0.90	C16-H16A	0.96
N3-C15	1.366(5)	C17-C18	1.373(6)
N3-C18	1.371(5)	C17-H17A	0.96
N3-H3N	0.90	C18-C19	1.434(6)
N4-C19	1.376(5)	C19-C20	1.355(6)
N4-C22	1.377(5)	C20-C21	1.423(6)
N4-H4N	0.90	C20-H20A	0.96

C21-C22	1.371(6)	C1A-H1AA	0.96
C21-H21A	0.96	C1A-H1AB	0.96
C22-C23	1.509(6)	C2A-C3AA	1.510(10)
C23-C34	1.505(6)	C2A-C3A	1.553(10)
C23-C33	1.585(6)	C2A-H2AC	0.96
C28-C35	1.525(6)	C2A-H2AD	0.96
C28-C36	1.538(6)	C2A-H2AA	0.96
C29-H29A	0.96	C2A-H2AB	0.96
C29-H29B	0.96	C3A-C4A	1.400(13)
C29-H29C	0.96	C3A-H3AA	0.96
C30-H30A	0.96	C3A-H3AB	0.96
C30-H30B	0.96	C4A-H4AA	0.96
C30-H30C	0.96	C4A-H4AB	0.96
C31-H31A	0.96	C4A-H4AC	0.96
C31-H31B	0.96	C3AA-C4AA	1.407(14)
C31-H31C	0.96	C3AA-H3AC	0.96
C32-H32A	0.96	C3AA-H3AD	0.96
C32-H32B	0.96	C4AA-H4AD	0.96
C32-H32C	0.96	C4AA-H4AE	0.96
C33-H33A	0.96	C4AA-H4AF	0.96
C33-H33B	0.96	C5A-C6A	1.501(7)
C33-H33C	0.96	C5A-H5AA	0.96
C34-H34A	0.96	C5A-H5AB	0.96
C34-H34B	0.96	C6A-C7A	1.495(7)
C34-H34C	0.96	C6A-H6AA	0.96
C35-H35A	0.96	C6A-H6AB	0.96
C35-H35B	0.96	C7A-C8A	1.502(8)
C35-H35C	0.96	C7A-H7AA	0.96
C36-H36A	0.96	C7A-H7AB	0.96
C36-H36B	0.96	C8A-H8AA	0.96
C36-H36C	0.96	C8A-H8AB	0.96
N1A-C1A	1.501(6)	C8A-H8AC	0.96
N1A-C5A	1.511(6)	C9A-C10A	1.470(6)
N1A-C13A	1.525(6)	C9A-H9AA	0.96
N1A-C9A	1.526(5)	C9A-H9AB	0.96
C1A-C2A	1.495(7)	C10A-C11A	1.498(6)

C10A-H10A	0.96	C13A-H13B	0.96
C10A-H10B	0.96	C14A-C15A	1.516(7)
C11A-C12A	1.518(6)	C14A-H14A	0.96
C11A-H11B	0.96	C14A-H14B	0.96
C11A-H11C	0.96	C15A-C16A	1.525(7)
C12A-H12B	0.96	C15A-H15A	0.96
C12A-H12C	0.96	C15A-H15B	0.96
C12A-H12D	0.96	C16A-H16B	0.96
C13A-C14A	1.514(6)	C16A-H16C	0.96
C13A-H13A	0.96	C16A-H16D	0.96
C27-S2-C24	96.0(3)	C23-C24A-S2A	125.6(10)
C25-C24-C23	127.9(6)	C13-S1-C10	93.4(2)
C25-C24-S2	109.0(4)	H1WA-O1W-H1WB	114(7)
C23-C24-S2	123.1(5)	C4-N1-C1	110.2(4)
C24-C25-C26	112.8(6)	C4-N1-H1N	124.8
C24-C25-H25	123.0	C1-N1-H1N	125.0
C26-C25-H25	124.2	C8-N2-C5	110.3(4)
C27-C26-C25	113.7(5)	C8-N2-H2N	124.7
C27-C26-H26	122.6	C5-N2-H2N	125.0
C25-C26-H26	123.7	C15-N3-C18	111.0(4)
C26-C27-C28	126.0(5)	C15-N3-H3N	124.4
C26-C27-S2	108.5(4)	C18-N3-H3N	124.6
C28-C27-S2	125.5(4)	C19-N4-C22	110.7(4)
C27A-S2A-C24A	99.9(8)	C19-N4-H4N	124.7
C28-C27A-S2A	128.6(11)	C22-N4-H4N	124.7
C28-C27A-C26A	112.4(11)	C2-C1-N1	105.6(4)
S2A-C27A-C26A	100.0(11)	C2-C1-C28	132.6(4)
C25A-C26A-C27A	111(2)	N1-C1-C28	121.8(4)
C25A-C26A-H26A	125.1	C1-C2-C3	108.6(4)
C27A-C26A-H26A	124.0	C1-C2-H2A	125.7
C24A-C25A-C26A	110(2)	C3-C2-H2A	125.7
C24A-C25A-H25A	124.7	C4-C3-C2	108.7(4)
C26A-C25A-H25A	125.2	C4-C3-H3A	125.4
C25A-C24A-C23	121.3(13)	C2-C3-H3A	125.8
C25A-C24A-S2A	113.0(13)	C3-C4-N1	106.8(4)

C3-C4-C5	130.4(5)	C15-C14-C32	108.4(4)
N1-C4-C5	122.8(4)	C31-C14-C32	108.3(4)
C6-C5-N2	107.8(4)	N3-C15-C16	107.2(4)
C6-C5-C4	128.2(5)	N3-C15-C14	123.3(4)
N2-C5-C4	124.0(4)	C16-C15-C14	129.5(5)
C5-C6-C7	106.5(5)	C15-C16-C17	106.5(4)
C5-C6-H6A	126.7	C15-C16-H16A	126.7
C7-C6-H6A	126.8	C17-C16-H16A	126.8
C8-C7-C6	108.0(4)	C18-C17-C16	109.9(5)
C8-C7-H7A	126.2	C18-C17-H17A	125.2
C6-C7-H7A	125.9	C16-C17-H17A	125.0
N2-C8-C7	107.4(4)	N3-C18-C17	105.4(4)
N2-C8-C9	122.0(4)	N3-C18-C19	126.4(4)
C7-C8-C9	130.5(4)	C17-C18-C19	128.1(5)
C10-C9-C8	109.9(4)	C20-C19-N4	106.9(4)
C10-C9-C29	109.9(4)	C20-C19-C18	128.9(5)
C8-C9-C29	108.9(4)	N4-C19-C18	124.2(4)
C10-C9-C30	110.9(4)	C19-C20-C21	108.2(4)
C8-C9-C30	108.8(4)	C19-C20-H20A	126.1
C29-C9-C30	108.4(4)	C21-C20-H20A	125.6
C11-C10-C9	130.2(4)	C22-C21-C20	107.7(5)
C11-C10-S1	108.0(4)	C22-C21-H21A	126.1
C9-C10-S1	121.8(3)	C20-C21-H21A	126.2
C10-C11-C12	115.5(4)	C21-C22-N4	106.5(4)
C10-C11-H11A	122.3	C21-C22-C23	130.7(5)
C12-C11-H11A	122.1	N4-C22-C23	122.6(4)
C13-C12-C11	113.1(4)	C34-C23-C22	110.2(4)
C13-C12-H12A	123.4	C34-C23-C24	113.3(4)
C11-C12-H12A	123.5	C22-C23-C24	110.3(4)
C12-C13-C14	128.5(4)	C34-C23-C24A	93.1(6)
C12-C13-S1	110.0(4)	C22-C23-C24A	111.4(6)
C14-C13-S1	121.3(3)	C34-C23-C33	107.4(4)
C13-C14-C15	109.6(4)	C22-C23-C33	108.8(4)
C13-C14-C31	110.3(4)	C24-C23-C33	106.6(5)
C15-C14-C31	110.3(4)	C24A-C23-C33	124.3(6)
C13-C14-C32	109.8(4)	C1-C28-C27	110.8(4)

C1-C28-C27A	118.7(7)	H33A-C33-H33C	109.5
C1-C28-C35	109.9(4)	H33B-C33-H33C	109.5
C27-C28-C35	108.1(4)	C23-C34-H34A	109.9
C27A-C28-C35	108.0(6)	C23-C34-H34B	108.8
C1-C28-C36	109.1(4)	H34A-C34-H34B	109.5
C27-C28-C36	111.0(5)	C23-C34-H34C	109.7
C27A-C28-C36	102.6(7)	H34A-C34-H34C	109.5
C35-C28-C36	107.9(4)	H34B-C34-H34C	109.5
C9-C29-H29A	109.4	C28-C35-H35A	109.1
C9-C29-H29B	109.5	C28-C35-H35B	109.6
H29A-C29-H29B	109.5	H35A-C35-H35B	109.5
C9-C29-H29C	109.5	C28-C35-H35C	109.7
H29A-C29-H29C	109.5	H35A-C35-H35C	109.5
H29B-C29-H29C	109.5	H35B-C35-H35C	109.5
C9-C30-H30A	109.3	C28-C36-H36A	109.4
C9-C30-H30B	110.0	C28-C36-H36B	109.2
H30A-C30-H30B	109.5	H36A-C36-H36B	109.5
C9-C30-H30C	109.1	C28-C36-H36C	109.8
H30A-C30-H30C	109.5	H36A-C36-H36C	109.5
H30B-C30-H30C	109.5	H36B-C36-H36C	109.5
C14-C31-H31A	109.6	C1A-N1A-C5A	108.2(4)
C14-C31-H31B	109.4	C1A-N1A-C13A	110.6(4)
H31A-C31-H31B	109.5	C5A-N1A-C13A	109.6(4)
C14-C31-H31C	109.4	C1A-N1A-C9A	110.2(4)
H31A-C31-H31C	109.5	C5A-N1A-C9A	110.6(4)
H31B-C31-H31C	109.5	C13A-N1A-C9A	107.7(4)
C14-C32-H32A	109.5	C2A-C1A-N1A	116.3(5)
C14-C32-H32B	109.3	C2A-C1A-H1AA	107.7
H32A-C32-H32B	109.5	N1A-C1A-H1AA	108.6
C14-C32-H32C	109.7	C2A-C1A-H1AB	107.1
H32A-C32-H32C	109.5	N1A-C1A-H1AB	109.0
H32B-C32-H32C	109.5	H1AA-C1A-H1AB	107.8
C23-C33-H33A	109.0	C1A-C2A-C3AA	114.1(7)
C23-C33-H33B	109.7	C1A-C2A-C3A	108.7(6)
H33A-C33-H33B	109.5	C1A-C2A-H2AC	109.1
C23-C33-H33C	109.7	C3AA-C2A-H2AC	108.7

C1A-C2A-H2AD	107.9	H4AE-C4AA-H4AF	109.5
C3AA-C2A-H2AD	108.4	C6A-C5A-N1A	116.1(4)
H2AC-C2A-H2AD	108.4	C6A-C5A-H5AA	108.5
C1A-C2A-H2AA	108.6	N1A-C5A-H5AA	108.3
C3A-C2A-H2AA	108.9	C6A-C5A-H5AB	107.9
C1A-C2A-H2AB	109.6	N1A-C5A-H5AB	108.3
C3A-C2A-H2AB	112.2	H5AA-C5A-H5AB	107.4
H2AA-C2A-H2AB	108.8	C7A-C6A-C5A	111.2(5)
C4A-C3A-C2A	110.5(10)	C7A-C6A-H6AA	109.3
C4A-C3A-H3AA	110.2	C5A-C6A-H6AA	109.8
C2A-C3A-H3AA	108.4	C7A-C6A-H6AB	109.4
C4A-C3A-H3AB	107.8	C5A-C6A-H6AB	109.1
C2A-C3A-H3AB	111.6	H6AA-C6A-H6AB	108.1
H3AA-C3A-H3AB	108.3	C6A-C7A-C8A	117.6(6)
C3A-C4A-H4AA	111.1	C6A-C7A-H7AA	108.0
C3A-C4A-H4AB	107.4	C8A-C7A-H7AA	107.8
H4AA-C4A-H4AB	109.5	C6A-C7A-H7AB	107.9
C3A-C4A-H4AC	109.8	C8A-C7A-H7AB	107.9
H4AA-C4A-H4AC	109.5	H7AA-C7A-H7AB	107.3
H4AB-C4A-H4AC	109.5	C7A-C8A-H8AA	109.7
H4AC-C4A-H3AC	113.9	C7A-C8A-H8AB	109.3
C4AA-C3AA-C2A	113.6(11)	H8AA-C8A-H8AB	109.5
C2A-C3AA-H3AA	111.1	C7A-C8A-H8AC	109.4
C4AA-C3AA-H4AB	111.6	H8AA-C8A-H8AC	109.5
C2A-C3AA-H4AB	118.2	H8AB-C8A-H8AC	109.5
C4AA-C3AA-H3AC	108.9	C10A-C9A-N1A	117.9(4)
C2A-C3AA-H3AC	112.4	C10A-C9A-H9AA	107.6
C4AA-C3AA-H3AD	106.0	N1A-C9A-H9AA	107.6
C2A-C3AA-H3AD	107.6	C10A-C9A-H9AB	108.3
H3AC-C3AA-H3AD	108.1	N1A-C9A-H9AB	107.8
H3AA-C4AA-H3AB	109.0	H9AA-C9A-H9AB	107.2
C3AA-C4AA-H4AD	113.5	C9A-C10A-C11A	111.4(4)
C3AA-C4AA-H4AE	105.9	C9A-C10A-H10A	109.7
H4AD-C4AA-H4AE	109.5	C11A-C10A-H10A	109.5
C3AA-C4AA-H4AF	108.9	C9A-C10A-H10B	109.0
H4AD-C4AA-H4AF	109.5	C11A-C10A-H10B	109.3

H10A-C10A-H10B	107.9	C13A-C14A-C15A	109.8(4)
C10A-C11A-C12A	113.8(4)	C13A-C14A-H14A	109.7
C10A-C11A-H11B	109.0	C15A-C14A-H14A	109.8
C12A-C11A-H11B	108.7	C13A-C14A-H14B	109.6
C10A-C11A-H11C	108.8	C15A-C14A-H14B	109.6
C12A-C11A-H11C	108.7	H14A-C14A-H14B	108.3
H11B-C11A-H11C	107.7	C14A-C15A-C16A	114.1(5)
C11A-C12A-H12B	109.7	C14A-C15A-H15A	108.4
C11A-C12A-H12C	109.3	C16A-C15A-H15A	108.6
H12B-C12A-H12C	109.5	C14A-C15A-H15B	109.3
C11A-C12A-H12D	109.4	C16A-C15A-H15B	108.7
H12B-C12A-H12D	109.5	H15A-C15A-H15B	107.7
H12C-C12A-H12D	109.5	C15A-C16A-H16B	109.7
C14A-C13A-N1A	116.7(4)	C15A-C16A-H16C	109.3
C14A-C13A-H13A	108.0	H16B-C16A-H16C	109.5
N1A-C13A-H13A	108.1	C15A-C16A-H16D	109.5
C14A-C13A-H13B	107.8	H16B-C16A-H16D	109.5
N1A-C13A-H13B	108.4	H16C-C16A-H16D	109.5
H13A-C13A-H13B	107.4		

Table 4. Anisotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for $7 \cdot (\text{C}_4\text{H}_9)_4\text{NCl} \cdot \text{H}_2\text{O} \cdot 2\text{CH}_2\text{Cl}_2$. The anisotropic displacement factor exponent takes the form: $-2\pi^2 [h^2 a^{*2} U^{11} + \dots + 2 h k a^* b^* U^{12}]$.

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
S2	60(1)	33(1)	45(1)	4(1)	17(1)	4(1)
C24	44(4)	44(4)	24(4)	-4(4)	2(3)	5(4)
C25	76(5)	49(5)	54(5)	3(4)	28(4)	-27(4)
C26	72(5)	24(4)	60(5)	-6(4)	17(4)	-1(4)
C27	36(5)	58(7)	35(6)	-8(4)	-10(4)	12(4)
S2A	27(4)	44(4)	69(5)	-13(4)	9(3)	-14(4)
S1	48(1)	36(1)	38(1)	7(1)	2(1)	7(1)
Cl1	52(1)	36(1)	56(1)	6(1)	3(1)	0(1)
O1W	42(2)	50(3)	38(2)	2(2)	12(2)	-17(2)
N1	35(2)	27(2)	42(3)	-1(2)	8(2)	-5(2)
N2	45(2)	21(2)	37(2)	4(2)	8(2)	-3(2)
N3	34(2)	30(2)	33(2)	8(2)	-1(2)	8(2)
N4	45(2)	23(2)	45(3)	2(2)	7(2)	-1(2)
C1	36(3)	26(3)	44(3)	-3(3)	9(3)	5(2)
C2	44(3)	34(3)	63(4)	0(3)	24(3)	-10(3)
C3	51(3)	42(3)	32(3)	5(3)	17(3)	-8(3)
C4	40(3)	29(3)	32(3)	0(2)	9(3)	3(2)
C5	46(3)	30(3)	37(3)	5(2)	12(3)	6(3)
C6	56(3)	64(4)	38(3)	-1(3)	15(3)	4(3)
C7	57(4)	59(4)	44(4)	2(3)	1(3)	1(3)
C8	39(3)	30(3)	35(3)	2(3)	-2(3)	-4(2)
C9	40(3)	27(3)	41(3)	-3(3)	-6(3)	2(2)
C10	29(3)	29(3)	43(3)	4(3)	-6(2)	-2(2)
C11	42(3)	30(3)	42(3)	13(3)	-7(3)	6(2)
C12	47(3)	37(3)	30(3)	8(3)	4(3)	-2(3)
C13	35(3)	33(3)	42(3)	6(3)	3(2)	-3(2)
C14	35(3)	46(3)	46(3)	5(3)	15(3)	5(3)
C15	42(3)	35(3)	37(3)	-4(3)	13(3)	7(3)
C16	49(3)	41(3)	51(3)	7(3)	2(3)	14(3)

C17	62(4)	31(3)	61(4)	4(3)	2(3)	3(3)
C18	46(3)	27(3)	36(3)	1(2)	3(3)	3(3)
C19	44(3)	26(3)	33(3)	1(2)	13(3)	0(3)
C20	65(4)	30(3)	45(3)	1(3)	2(3)	0(3)
C21	63(4)	34(3)	56(4)	-7(3)	9(3)	-10(3)
C22	52(3)	29(3)	37(3)	-10(3)	8(3)	-1(3)
C23	47(3)	45(3)	36(3)	-5(3)	4(3)	2(3)
C28	31(3)	28(3)	51(3)	0(3)	7(2)	0(2)
C29	51(3)	32(3)	53(3)	0(3)	0(3)	-11(3)
C30	55(3)	37(3)	58(4)	0(3)	-6(3)	5(3)
C31	44(3)	58(3)	52(3)	-11(3)	5(3)	14(3)
C32	51(3)	64(4)	61(4)	3(3)	16(3)	-1(3)
C33	85(4)	107(5)	59(4)	16(4)	1(3)	15(4)
C34	86(4)	62(4)	89(4)	-16(3)	-28(4)	-16(3)
C35	86(4)	47(4)	78(4)	10(3)	-1(3)	-4(3)
C36	46(3)	112(5)	85(4)	11(4)	8(3)	-7(3)
N1A	59(3)	41(3)	42(3)	-1(2)	4(2)	-23(2)
C1A	78(4)	59(4)	58(4)	-1(3)	9(3)	-29(3)
C2A	79(4)	84(5)	84(5)	-13(4)	-13(4)	-22(4)
C3A	140(11)	133(11)	141(11)	26(9)	20(9)	-39(8)
C4A	108(8)	86(7)	163(9)	2(7)	-1(7)	-18(7)
C3AA	58(8)	78(9)	66(9)	14(7)	-23(8)	-35(7)
C4AA	133(12)	147(12)	143(12)	-33(9)	35(9)	-2(9)
C5A	77(4)	34(3)	62(4)	6(3)	16(3)	-4(3)
C6A	116(5)	59(4)	81(5)	-7(4)	28(4)	18(4)
C7A	115(5)	77(5)	115(5)	15(4)	54(4)	20(4)
C8A	170(6)	101(5)	151(6)	5(5)	68(5)	49(5)
C9A	52(3)	37(3)	39(3)	7(3)	12(3)	-9(3)
C10A	52(3)	42(3)	59(4)	9(3)	13(3)	-6(3)
C11A	57(3)	29(3)	55(3)	-2(3)	22(3)	-4(3)
C12A	61(4)	55(4)	95(4)	-12(3)	33(3)	-11(3)
C13A	63(3)	45(3)	53(3)	-1(3)	16(3)	-20(3)
C14A	78(4)	58(4)	74(4)	-1(3)	35(3)	-21(3)
C15A	88(4)	101(5)	73(4)	1(4)	39(4)	-31(4)
C16A	100(5)	78(5)	109(5)	-29(4)	54(4)	-39(4)

Table 5. Hydrogen coordinates ($\times 10^4$) and isotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for $7 \cdot (\text{C}_4\text{H}_9)_4\text{NCl} \cdot \text{H}_2\text{O} \cdot 2\text{CH}_2\text{Cl}_2$.

	x	y	z	U(eq)
H25	3011	3630	-1080	69
H26	2277	4488	-431	62
H26A	518	3015	-122	89
H25A	1203	2251	-915	101
H1N	3190	4470	1274	41
H2N	4612	4748	1794	41
H3N	6150	2933	362	40
H4N	4650	2617	-142	45
H2A	1249	3374	1903	55
H3A	2519	3516	2820	49
H6A	4062	4038	3492	62
H7A	5631	4599	3661	65
H11A	6059	5293	1107	47
H12A	6759	4423	476	45
H16A	7744	1835	1705	57
H17A	6412	1075	1275	63
H20A	4995	626	498	57
H21A	3471	717	-209	61
H29A	5458	5903	1878	70
H29B	6411	6194	2150	70
H29C	5668	6182	2593	70
H30A	7104	4783	3245	77
H30B	6677	5502	3430	77
H30C	7417	5511	2984	77
H31A	8481	3006	1992	78
H31B	9016	3514	1596	78
H31C	8933	2698	1425	78
H32A	7514	3490	111	87

H32B	8347	2991	285	87
H32C	8426	3811	442	87
H33A	3796	2002	-1548	127
H33B	3786	2802	-1323	127
H33C	2951	2477	-1763	127
H34A	2068	1431	-638	125
H34B	2568	1199	-1206	125
H34C	1859	1814	-1312	125
H35A	2097	5031	541	108
H35B	1183	5101	796	108
H35C	1201	4915	67	108
H36A	578	3285	739	122
H36B	252	3865	213	122
H36C	286	4043	949	122
H1AA	1670	542	1735	78
H1AB	2145	721	1149	78
H2AC	1430	1783	997	102
H2AD	1029	1679	1633	102
H2AA	1469	1873	1152	102
H2AB	851	1535	1601	102
H3AA	526	616	739	166
H3AB	946	1133	287	166
H4AA	-530	1272	126	182
H4AB	-474	1452	864	182
H4AC	-50	1972	408	182
H3AC	-124	1396	840	85
H3AD	255	670	1122	85
H4AD	9	602	41	209
H4AE	641	1264	36	209
H4AF	1024	532	321	209
H5AA	3768	793	2552	68
H5AB	2987	257	2396	68
H6AA	3355	44	1385	101
H6AB	4108	613	1509	101
H7AA	4087	-651	2211	118
H7AB	4828	-81	2354	118

H8AA	5352	-1026	1829	204
H8AB	5308	-352	1379	204
H8AC	4568	-943	1250	204
H9AA	3309	1575	1229	51
H9AB	2786	2175	1511	51
H10A	4466	1698	2047	60
H10B	3934	2258	2381	60
H11B	3954	3041	1527	55
H11C	4446	2477	1169	55
H12B	5460	3328	1638	102
H12C	5706	2584	1964	102
H12D	5208	3158	2321	102
H13A	2054	2008	2390	63
H13B	2897	1745	2841	63
H14A	2118	717	3078	81
H14B	1267	1017	2651	81
H15A	2141	1729	3751	101
H15B	1294	1272	3766	101
H16B	911	2481	3691	137
H16C	453	2082	3068	137
H16D	1310	2543	3046	137
H1WA	4190(30)	5240(30)	730(30)	70(20)
H1WB	4510(50)	4620(40)	610(40)	160(50)

Table 6. Torsion angles [°] for **7**·(C₄H₉)₄NCl·H₂O·2CH₂Cl₂.

C27-S2-C24-C25	1.2(4)	C4-C5-C6-C7	-179.3(4)
C27-S2-C24-C23	-177.5(5)	C5-C6-C7-C8	-0.2(6)
C23-C24-C25-C26	177.2(5)	C5-N2-C8-C7	-0.8(5)
S2-C24-C25-C26	-1.4(5)	C5-N2-C8-C9	-178.0(4)
C24-C25-C26-C27	1.0(8)	C6-C7-C8-N2	0.6(5)
C25-C26-C27-C28	179.4(6)	C6-C7-C8-C9	177.5(5)
C25-C26-C27-S2	-0.1(9)	N2-C8-C9-C10	-56.8(5)
C24-S2-C27-C26	-0.6(6)	C7-C8-C9-C10	126.7(5)
C24-S2-C27-C28	179.9(7)	N2-C8-C9-C29	63.6(5)
C24A-S2A-C27A-C28	149.6(10)	C7-C8-C9-C29	-112.9(5)
C24A-S2A-C27A-C26A	20.2(9)	N2-C8-C9-C30	-178.4(4)
C28-C27A-C26A-C25A	-164.4(11)	C7-C8-C9-C30	5.1(7)
S2A-C27A-C26A-C25A	-25.2(14)	C8-C9-C10-C11	97.9(5)
C27A-C26A-C25A-C24A	18.4(14)	C29-C9-C10-C11	-21.8(6)
C26A-C25A-C24A-C23	-179.3(12)	C30-C9-C10-C11	-141.7(5)
C26A-C25A-C24A-S2A	-3.2(9)	C8-C9-C10-S1	-79.6(4)
C27A-S2A-C24A-C25A	-12.6(5)	C29-C9-C10-S1	160.6(3)
C27A-S2A-C24A-C23	163.2(9)	C30-C9-C10-S1	40.7(5)
C4-N1-C1-C2	0.0(5)	C13-S1-C10-C11	-0.4(3)
C4-N1-C1-C28	178.1(4)	C13-S1-C10-C9	177.6(4)
N1-C1-C2-C3	0.0(5)	C9-C10-C11-C12	-177.0(4)
C28-C1-C2-C3	-177.8(5)	S1-C10-C11-C12	0.8(5)
C1-C2-C3-C4	0.0(5)	C10-C11-C12-C13	-0.9(6)
C2-C3-C4-N1	0.0(5)	C11-C12-C13-C14	175.9(4)
C2-C3-C4-C5	178.9(4)	C11-C12-C13-S1	0.5(5)
C1-N1-C4-C3	0.0(5)	C10-S1-C13-C12	0.0(3)
C1-N1-C4-C5	-179.0(4)	C10-S1-C13-C14	-175.8(4)
C8-N2-C5-C6	0.7(5)	C12-C13-C14-C15	-92.7(5)
C8-N2-C5-C4	179.7(4)	S1-C13-C14-C15	82.2(4)
C3-C4-C5-C6	-5.8(8)	C12-C13-C14-C31	145.7(5)
N1-C4-C5-C6	173.0(4)	S1-C13-C14-C31	-39.4(5)
C3-C4-C5-N2	175.4(4)	C12-C13-C14-C32	26.3(6)
N1-C4-C5-N2	-5.8(7)	S1-C13-C14-C32	-158.7(3)
N2-C5-C6-C7	-0.3(5)	C18-N3-C15-C16	-1.1(5)

C18-N3-C15-C14	-179.5(4)	S2-C24-C23-C34	59.9(5)
C13-C14-C15-N3	60.6(5)	C25-C24-C23-C22	117.4(5)
C31-C14-C15-N3	-177.7(4)	S2-C24-C23-C22	-64.2(5)
C32-C14-C15-N3	-59.3(5)	C25-C24-C23-C24A	-145.4(15)
C13-C14-C15-C16	-117.4(5)	S2-C24-C23-C24A	33.0(13)
C31-C14-C15-C16	4.2(7)	C25-C24-C23-C33	-0.6(6)
C32-C14-C15-C16	122.7(5)	S2-C24-C23-C33	177.8(3)
N3-C15-C16-C17	2.3(5)	C25A-C24A-C23-C34	-21.4(6)
C14-C15-C16-C17	-179.5(4)	S2A-C24A-C23-C34	163.1(8)
C15-C16-C17-C18	-2.7(6)	C25A-C24A-C23-C22	-134.5(6)
C15-N3-C18-C17	-0.6(5)	S2A-C24A-C23-C22	50.0(10)
C15-N3-C18-C19	-177.6(4)	C25A-C24A-C23-C24	134.1(14)
C16-C17-C18-N3	2.0(5)	S2A-C24A-C23-C24	-41.4(10)
C16-C17-C18-C19	178.9(5)	C25A-C24A-C23-C33	92.1(8)
C22-N4-C19-C20	2.2(5)	S2A-C24A-C23-C33	-83.4(10)
C22-N4-C19-C18	-175.7(4)	C2-C1-C28-C27	-116.5(6)
N3-C18-C19-C20	170.3(5)	N1-C1-C28-C27	66.0(6)
C17-C18-C19-C20	-6.0(8)	C2-C1-C28-C27A	-111.0(8)
N3-C18-C19-N4	-12.3(7)	N1-C1-C28-C27A	71.5(8)
C17-C18-C19-N4	171.4(5)	C2-C1-C28-C35	124.1(5)
N4-C19-C20-C21	-2.1(5)	N1-C1-C28-C35	-53.4(5)
C18-C19-C20-C21	175.7(4)	C2-C1-C28-C36	6.0(7)
C19-C20-C21-C22	1.3(6)	N1-C1-C28-C36	-171.5(4)
C20-C21-C22-N4	0.1(5)	C26-C27-C28-C1	-120.7(8)
C20-C21-C22-C23	175.2(4)	S2-C27-C28-C1	58.7(8)
C19-N4-C22-C21	-1.4(5)	C26-C27-C28-C27A	90(3)
C19-N4-C22-C23	-177.0(4)	S2-C27-C28-C27A	-90(3)
C21-C22-C23-C34	3.3(7)	C26-C27-C28-C35	-0.2(10)
N4-C22-C23-C34	177.8(4)	S2-C27-C28-C35	179.2(6)
C21-C22-C23-C24	129.2(6)	C26-C27-C28-C36	118.0(8)
N4-C22-C23-C24	-56.4(6)	S2-C27-C28-C36	-62.6(8)
C21-C22-C23-C24A	105.2(7)	S2A-C27A-C28-C1	-12.7(14)
N4-C22-C23-C24A	-80.4(7)	C26A-C27A-C28-C1	111.9(11)
C21-C22-C23-C33	-114.2(6)	S2A-C27A-C28-C27	21(2)
N4-C22-C23-C33	60.3(6)	C26A-C27A-C28-C27	145(3)
C25-C24-C23-C34	-118.5(5)	S2A-C27A-C28-C35	113.1(10)

C26A-C27A-C28-C35	-122.2(10)	N1A-C5A-C6A-C7A	176.6(5)
S2A-C27A-C28-C36	-133.1(10)	C5A-C6A-C7A-C8A	-178.0(6)
C26A-C27A-C28-C36	-8.4(12)	C1A-N1A-C9A-C10A	-172.5(4)
C5A-N1A-C1A-C2A	175.4(5)	C5A-N1A-C9A-C10A	-52.9(6)
C13A-N1A-C1A-C2A	55.4(6)	C13A-N1A-C9A-C10A	66.9(5)
C9A-N1A-C1A-C2A	-63.5(6)	N1A-C9A-C10A-C11A	-175.2(4)
N1A-C1A-C2A-C3AA	-172.1(8)	C9A-C10A-C11A-C12A	-177.8(4)
N1A-C1A-C2A-C3A	161.5(8)	C1A-N1A-C13A-C14A	54.1(6)
C1A-C2A-C3A-C4A	165.5(11)	C5A-N1A-C13A-C14A	-65.1(5)
C1A-C2A-C3AA-C4AA	-72.5(16)	C9A-N1A-C13A-C14A	174.5(4)
C1A-N1A-C5A-C6A	66.7(5)	N1A-C13A-C14A-C15A	177.0(5)
C13A-N1A-C5A-C6A	-172.7(4)	C13A-C14A-C15A-C16A	65.5(6)
C9A-N1A-C5A-C6A	-54.1(6)		

Table 7. Hydrogen bonds for **7**·(C₄H₉)₄NCl·H₂O·2CH₂Cl₂ [Å and °].

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)
N1-H1N...O1W	0.90	2.07	2.939(5)	162.8
N2-H2N...O1W	0.90	2.16	3.032(5)	164.3
N3-H3N...Cl1	0.90	2.43	3.300(4)	161.9
N4-H4N...Cl1	0.90	2.31	3.207(4)	171.5
O1W-H1WB...Cl1	0.70(8)	2.66(8)	3.312(6)	156(8)
O1W-H1WA...Cl1#1	0.83(6)	2.41(6)	3.125(5)	143(5)

Symmetry transformations used to generate equivalent atoms:

#1 -x+1,-y+1,-z

Figure 1. View of the macrocycle in **7**·(C₄H₉)₄NCl·H₂O·2CH₂Cl₂ showing the disorder of one of the thiophene moieties. Displacement ellipsoids are scaled to the 40% probability level. Most hydrogen atoms have been removed for clarity.

Figure 2. View of the cation in $7 \cdot (\text{C}_4\text{H}_9)_4\text{NCl} \cdot \text{H}_2\text{O} \cdot 2\text{CH}_2\text{Cl}_2$ showing the atom labeling scheme. Displacement ellipsoids are scaled to the 40% probability level. The H atoms have been removed for clarity. One of the n-butyl side arms has some disorder as shown.