C1—C6	1.484 (5)	N4—C27	1.143 (5)
C2—C3	1.354 (4)	N5—C29	1.130 (5)
C3—C5	1.502 (4)	O1—C28	1.223 (4)
C6—C7	1.389 (3)	C19—C20	1.417 (4)
C6—C8	1.410 (5)	C19—C24	1.422 (4)
C7—C8	1.431 (5)	C19—C25	1.412 (4)
C7—C12	1.437 (5)	C20—C21	1.350 (5)
C8—C9	1.423 (4)	C21—C22	1.415 (5)
C9—C10	1.347 (6)	C22—C23	1.397 (4)
C10—C11	1.412 (6)	C22—C28	1.429 (4)
C11—C12	1.352 (4)	C23—C24	1.361 (4)
N1—C17	1.133 (5)	C25—C26	1.403 (4)
N2—C18	1.141 (5)	C25—C27	1.403 (5)
C13—C14	1.446 (4)	C28—C29	1.492 (5)
C1—S1—C3	97.6 (2)	C15—C13—C16	121.2 (3)
C1—S2—C2	96.7 (2)	C13—C14—C15	120.7 (3)
C2—S3—C4	103.1 (2)	C13—C15—C14	121.5 (3)
S1—C1—S2	115.1 (2)	C13—C16—C17	122.0 (3)
S1—C1—C6	121.6 (2)	C13—C16—C18	122.6 (3)
S2—C1—C6	123.3 (2)	C17—C16—C18	115.5 (3)
S2—C2—S3	123.3 (2)	N1—C17—C16	178.9 (4)
S2—C2—C3	115.7 (3)	N2—C18—C16	179.6 (4)
S3—C2—C3	120.9 (3)	C20—C19—C24	116.9 (3)
S1—C3—C2	114.8 (2)	C20—C19—C25	121.6 (3)
S1—C3—C5	120.0 (2)	C24—C19—C25	121.5 (3)
C2—C3—C5	125.1 (3)	C19—C20—C21	121.4 (3)
C1—C6—C7	120.3 (3)	C20—C21—C22	121.4 (3)
C1—C6—C8	117.7 (2)	C21—C22—C23	117.7 (3)
C7—C6—C8	122.0 (3)	C21—C22—C28	120.3 (3)
C6—C7—C8	119.3 (3)	C23—C22—C28	122.0 (3)
C6—C7—C12	122.7 (3)	C22—C23—C24	121.5 (3)
C8—C7—C12	118.0 (2)	C19—C24—C23	121.1 (3)
C6—C8—C7	118.7 (2)	C19—C25—C26	120.5 (3)
C6—C8—C9	122.7 (3)	C19—C25—C27	122.8 (3)
C7—C8—C9	118.5 (3)	C26—C25—C27	116.8 (3)
C8—C9—C10	121.5 (4)	N3—C26—C25	178.2 (3)
C9—C10—C11	120.4 (3)	N4—C27—C25	179.0 (4)
C10—C11—C12	120.8 (4)	O1—C28—C22	127.5 (3)
C7—C12—C11	120.8 (3)	O1—C28—C29	116.4 (3)
C14—C13—C15	117.7 (2)	C22—C28—C29	116.2 (3)
C14—C13—C16	121.0 (3)	N5—C29—C28	175.2 (4)

The structure was solved by direct methods and successive difference Fourier syntheses. Full-matrix least-squares anisotropic (β_{ij}) refinement was performed on F. H atoms were included in the structure-factor calculations [C—H = 1 Å, B = 5 Å^3]. All calculations were performed on a MicroVAX 3100 computer using the *SDP* programs (B. A. Frenz & Associates, Inc., 1985).

Lists of structure factors, anisotropic thermal parameters and H-atom coordinates have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55936 (18 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: PA1032]

References

B. A. Frenz & Associates, Inc. (1985). *SDP Structure Determination Package*. College Station, Texas, USA, and Enraf–Nonius, Delft, The Netherlands.

Bryce, M. R. & Moore, A. J. (1988). *Synth. Met.* **25**, 203–205.

Bryce, M. R., Moore, A. J., Lorcy, D., Dhindsa, A. S. & Robert, A. (1990). *J. Chem. Soc. Chem. Commun.* pp. 470–472.

Bryce, M. R., Moore, A. J., Masihul, H., Ashwell, G. J., Fraser, A. T., Clegg, W., Hursthouse, M. B. & Karaulov, A. I. (1990). *Angew. Chem. Int. Ed. Engl.* **29**, 1450–1452.

Suchanski, M. R. & Van Duyne, R. P. (1976). *J. Am. Chem. Soc.* **98**, 250–252.

Triki, S., Ouahab, L., Grandjean, D. & Fabre, J. M. (1991). *Acta Cryst.* C**47**, 1371–1373.

Walker, N. & Stuart, D. (1983). *Acta Cryst.* A**39**, 158–166.

Yakushi, K., Nishimura, S., Sugano, T. & Kuroda, H. (1980). *Acta Cryst.* B**36**, 358–363.

Acta Cryst. (1993). C**49**, 1192–1195

Structures of the 1:1 and 2:1 Adducts of (+)-(*S*)-[2.2]Paracyclophane-4-carboxylic Acid and (−)-Ephedrine

PETER G. JONES AND DETLEV DÖRING

Institut für Anorganische und Analytische Chemie der Technischen Universität, Hagenring 30, W-3300 Braunschweig, Germany

THOMAS LAUE AND HENNING HOPF

Institut für Organische Chemie der Technischen Universität, Hagenring 30, W-3300 Braunschweig, Germany

(*Received* 22 *June* 1992; *accepted* 23 *December* 1992)

Abstract

(+)-(*S*)-[2.2]Paracyclophane-4-carboxylic acid forms two adducts with (−)-ephedrine {α-[1-(methylamino)ethyl]benzenemethanol}. The 1:1 adduct, (−)-[1-(α-hydroxybenzyl)ethyl]methylammonium tricyclo[8.2.2.$2^{4,7}$]hexadeca-4,6,10,12,13,15-hexaene-5-carboxylate, is an ephedrinium carboxylate, whereas the 2:1 adduct contains an additional undissociated carboxylic acid molecule. Both adducts are stabilized by extensive systems of hydrogen bonding, with contacts as short as 2.57 Å, which connect the residues into helices.

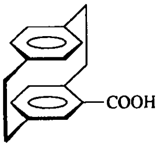
Comment

Substituted cyclophanes are chiral molecules. The first resolution of enantiomers was performed by Cram & Allinger (1955) with the brucine salt of [2.2]paracyclophane-4-carboxylic acid. Absolute configurations for this acid were established by Falk, Reich-Rohrwig & Schlögl (1970) and confirmed by X-ray analysis of a camphorate derivative by Tochtermann *et al.* (1987).

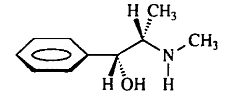
Since [2.2]paracyclophane derivatives racemize only under the most extreme conditions (Cram & Reich, 1969; Cram & Delton, 1970), it should be possible to use enantiomerically pure [2.2]paracyclophanes to separate enantiomers of racemic amines. However, attempts to resolve (±)-ephedrine with (+)-(*S*)-[2.2]paracyclophane-4-carboxylic acid (1)

0108-2701/93/061192-04$06.00

were not completely successful; fractional crystallization yielded salts of (−)-ephedrine (2) (confirmed by conversion to the hydrochloride and subsequent measurement of optical rotation), but the other diastereomeric salt could not be isolated with comparable purity even after repeated crystallizations. A further complication was provided by the fact that two different salts were formed, depending on the solvent system; a 1:1 salt was isolated pure from *tert*-butyl methyl ether and a 2:1 (cyclophane:base) adduct from isopropanol. Full details of the attempted separations are given by Laue (1991). Both the 1:1 and 2:1 adduct were subjected to X-ray analysis at 178 K.



(1)



(2)

The 1:1 *adduct*. Fig. 1 shows the asymmetric unit. Bond lengths and angles are normal. The cyclophane rings show the usual distortion (Vögtle, 1990), the bridgehead atoms lying 0.15–0.17 Å out of the plane of the remaining ring atoms. The carboxylate group is approximately coplanar with the ring [the dihedral angle between C(3), C(4), C(5) and C(17), O(1), O(2) is 10°].

H atoms were unambiguously located and confirm the adduct to be a salt of the carboxylate anion and the ephedrinium cation. The conformation of the cation is defined by the torsion angles 91, 180, −152° about the bonds [C(5′)—]C(4′)—C(1′), C(1′)—C(2′) and C(2′)—N′[—C(10′)], respectively.

Hydrogen bonds are formed within the asymmetric unit from the cation OH and NH_2 groups to carboxylate O atoms [O(3′)···O(1) 2.59, N′···O(2) 2.57 Å] and from the cation NH_2 to a symmetry-equivalent cation OH [N′···O(3′)($\frac{1}{2}+x$, $1\frac{1}{2}-y$, $1-z$) 2.88 Å]. The ions are thus linked in helices parallel to the *x* axis.

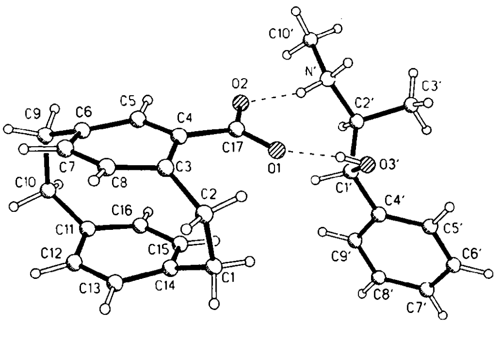


Fig. 1. The asymmetric unit of the 1:1 adduct. Radii are arbitrary. Hydrogen bonds are indicated by broken lines.

The 2:1 *adduct*. Fig. 2 shows the asymmetric unit. The conformations are similar to those of the 1:1 adduct, except that the carboxylate group C(17′), O(18′), O(19′) is rotated by 25° out of the plane C(3′), C(4′), C(5′).

Again, all H atoms could be located, showing that the asymmetric unit consists of one molecule of the carboxylic acid, one carboxylate anion (primed atom labels) and one ephedrinium cation (double-primed atom labels). The pattern of C—O bond lengths confirms this: C(17)—O(18) 1.209 (4), C(17)—O(19) 1.336 (3), C(17′)—O(18′) 1.256 (4), C(17′)—O(19′) 1.271 (5) Å.

Hydrogen bonds are formed within the asymmetric unit from the NH_2 group to a carboxylate O atom [N(11″)···O(18′) 2.79 Å]. Other hydrogen bonds are: NH_2 to carboxylate O atom [N(11″)···O(19′) 2.73 Å], hydroxyl to carboxylate O atom [O(12″)···O(18′) 2.74 Å] and carboxylic acid OH to carboxylate O atom [O(19)···O(19′) 2.61 Å] (symmetry operator of all second atoms: $1-x$, $-\frac{1}{2}+y$, $1-z$). The residues are thus connected by hydrophilic regions around the 2_1 screw axis.

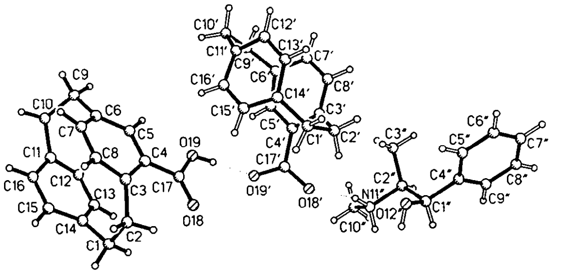


Fig. 2. The asymmetric unit of the 2:1 adduct. Radii are arbitrary. Hydrogen bonds are indicated by broken lines. The carboxylic acid molecule on the left is transformed by $1-x$, $\frac{1}{2}+y$, $1-z$ with respect to the coordinates in Table 2.

Experimental

1:1 adduct

Crystal data

$C_{10}H_{16}NO^+.C_{17}H_{15}NO_2^-$
$M_r = 417.5$
Orthorhombic
$P2_12_12_1$
$a = 7.736\ (2)$ Å
$b = 10.941\ (2)$ Å
$c = 26.445\ (5)$ Å
$V = 2238.4\ (8)$ Å^3
$Z = 4$
$D_x = 1.239$ Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.71069$ Å
Cell parameters from 49 reflections
$\theta = 10\text{-}12°$
$\mu = 0.08$ mm^{-1}
$T = 178$ K
Prism
0.85 × 0.85 × 0.4 mm
Colourless

Data collection

Siemens *R*3 diffractometer
ω scans
Absorption correction: none

$\theta_{\max} = 27.5°$
$h = -10 \rightarrow 0$
$k = -14 \rightarrow 0$
$l = -34 \rightarrow 0$

2956 measured reflections
2931 independent reflections
2497 observed reflections
$[F > 4\sigma(F)]$
3 standard reflections monitored every 147 reflections
intensity variation: ±1.5%

Refinement

Refinement on F
Final $R = 0.038$
$wR = 0.043$
$S = 1.5$
290 parameters
H atoms: O—H free, others riding
$w = 1/[\sigma^2(F) + 0.0003F^2]$
$(\Delta/\sigma)_{\max} = 0.006$
$\Delta\rho_{\max} = 0.17$ e Å^{-3}
$\Delta\rho_{\min} = -0.19$ e Å^{-3}
Atomic scattering factors from *SHELXTL-Plus*

Table 1. *Fractional atomic coordinates and equivalent isotropic thermal parameters* (Å^2) *for the* 1:1 *adduct*

$$U_{\rm eq} = \tfrac{1}{3}\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$U_{\rm eq}$
C(1′)	0.6737 (3)	0.5965 (2)	0.5501 (1)	0.223 (6)
C(2′)	0.4856 (3)	0.5573 (2)	0.5400 (1)	0.218 (6)
C(3′)	0.4487 (3)	0.5269 (2)	0.4849 (1)	0.291 (6)
C(4′)	0.7999 (3)	0.4973 (2)	0.5339 (1)	0.244 (6)
C(5′)	0.8697 (3)	0.4939 (2)	0.4854 (1)	0.305 (7)
C(6′)	0.9845 (3)	0.4025 (2)	0.4718 (1)	0.367 (7)
C(7′)	1.0313 (3)	0.3140 (2)	0.5066 (1)	0.405 (8)
C(8′)	0.9614 (3)	0.3156 (2)	0.5546 (1)	0.378 (7)
C(9′)	0.8458 (3)	0.4071 (2)	0.5681 (1)	0.297 (6)
C(10′)	0.1943 (3)	0.6160 (2)	0.5731 (1)	0.346 (7)
N′	0.3676 (2)	0.6585 (2)	0.5570 (1)	0.227 (5)
O(3′)	0.7080 (2)	0.7095 (1)	0.5250 (1)	0.279 (5)
C(1)	1.0234 (3)	0.9124 (3)	0.6616 (1)	0.459 (8)
C(2)	0.9028 (3)	1.0263 (2)	0.6522 (1)	0.330 (7)
C(3)	0.7405 (3)	1.0243 (2)	0.6841 (1)	0.247 (6)
C(4)	0.6070 (3)	0.9368 (2)	0.6802 (1)	0.228 (6)
C(5)	0.5097 (3)	0.9073 (2)	0.7230 (1)	0.240 (6)
C(6)	0.5405 (3)	0.9625 (2)	0.7697 (1)	0.272 (6)
C(7)	0.6426 (3)	1.0676 (2)	0.7694 (1)	0.319 (7)
C(8)	0.7391 (3)	1.0980 (2)	0.7273 (1)	0.287 (6)
C(9)	0.4973 (3)	0.8963 (3)	0.8180 (1)	0.386 (8)
C(10)	0.6423 (4)	0.8035 (3)	0.8355 (1)	0.485 (9)
C(11)	0.7921 (3)	0.7958 (2)	0.7988 (1)	0.332 (7)
C(12)	0.9265 (3)	0.8803 (2)	0.8001 (1)	0.339 (7)
C(13)	1.0218 (3)	0.9065 (2)	0.7573 (1)	0.322 (7)
C(14)	0.9842 (3)	0.8500 (2)	0.7114 (1)	0.326 (7)
C(15)	0.8773 (3)	0.7485 (2)	0.7133 (1)	0.354 (7)
C(16)	0.7823 (3)	0.7213 (2)	0.7564 (1)	0.361 (7)
C(17)	0.5801 (3)	0.8573 (2)	0.6340 (1)	0.234 (6)
O(1)	0.6782 (3)	0.8692 (2)	0.5969 (1)	0.406 (5)
O(2)	0.4575 (2)	0.7813 (2)	0.6354 (1)	0.386 (5)

2:1 adduct

Crystal data

$C_{10}H_{16}NO^+.C_{17}H_{15}NO_2^-.C_{17}H_{16}O_2$
$M_r = 669.8$
Monoclinic
$P2_1$
$a = 13.771$ (3) Å
$b = 7.788$ (2) Å
$c = 17.357$ (4) Å
$\beta = 106.39$ (2)°
$V = 1785.9$ (7) Å^3
$Z = 2$
$D_x = 1.246$ Mg m^{-3}
Mo $K\alpha$ radiation
$\lambda = 0.71069$ Å
Cell parameters from 48 reflections
$\theta = 10$–$11.5°$
$\mu = 0.08$ mm^{-1}
$T = 178$ K
Prism
0.6 × 0.45 × 0.3 mm
Colourless

Data collection

Siemens *R*3 diffractometer
ω scans
Absorption correction: none
5473 measured reflections
4391 independent reflections
3812 observed reflections
$[F > 4\sigma(F)]$
$R_{\rm int} = 0.016$
$\theta_{\max} = 27.5°$
$h = -17 \rightarrow 17$
$k = 0 \rightarrow 10$
$l = 0 \rightarrow 22$
3 standard reflections monitored every 147 reflections
intensity variation: ±1.5%

Refinement

Refinement on F
Final $R = 0.038$
$wR = 0.043$
$S = 1.5$
290 parameters
H atoms: riding
$w = 1/[\sigma^2(F) + 0.0003F^2]$
$(\Delta/\sigma)_{\max} = 0.006$
$\Delta\rho_{\max} = 0.17$ e Å^{-3}
$\Delta\rho_{\min} = -0.19$ e Å^{-3}
Atomic scattering factors from *SHELXTL-Plus*

Table 2. *Fractional atomic coordinates and equivalent isotropic thermal parameters* (Å^2) *for the* 2:1 *adduct*

$$U_{\rm eq} = \tfrac{1}{3}\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$U_{\rm eq}$
C(1)	0.4581 (2)	0.5000	0.0582 (2)	0.049 (1)
C(2)	0.3842 (2)	0.3426 (5)	0.0420 (2)	0.037 (1)
C(3)	0.2770 (2)	0.3946 (5)	0.0405 (1)	0.028 (1)
C(4)	0.2495 (2)	0.4539 (5)	0.1079 (1)	0.025 (1)
C(5)	0.1711 (2)	0.5739 (5)	0.0975 (1)	0.027 (1)
C(6)	0.1180 (2)	0.6336 (5)	0.0222 (1)	0.030 (1)
C(7)	0.1293 (2)	0.5406 (5)	−0.0434 (1)	0.034 (1)
C(8)	0.2070 (2)	0.4240 (5)	−0.0339 (1)	0.033 (1)
C(9)	0.0661 (2)	0.8067 (5)	0.0121 (2)	0.040 (1)
C(10)	0.1315 (2)	0.9548 (6)	−0.0096 (2)	0.054 (1)
C(11)	0.2411 (2)	0.9024 (5)	0.0048 (2)	0.043 (1)
C(12)	0.3047 (2)	0.8864 (5)	0.0823 (2)	0.042 (1)
C(13)	0.3849 (2)	0.7702 (5)	0.0983 (2)	0.039 (1)
C(14)	0.4039 (2)	0.6707 (5)	0.0383 (2)	0.038 (1)
C(15)	0.3541 (2)	0.7175 (6)	−0.0408 (2)	0.044 (1)
C(16)	0.2741 (2)	0.8307 (5)	−0.0569 (2)	0.044 (1)
C(17)	0.3073 (2)	0.4041 (5)	0.1908 (1)	0.028 (1)
O(18)	0.3824 (1)	0.3154 (5)	0.2085 (1)	0.051 (1)
O(19)	0.2670 (1)	0.4661 (5)	0.2467 (1)	0.033 (1)
C(1′)	0.8322 (2)	0.3287 (5)	0.6977 (1)	0.040 (1)
C(2′)	0.8000 (2)	0.3458 (5)	0.6028 (1)	0.033 (1)
C(3′)	0.8333 (2)	0.5149 (5)	0.5749 (1)	0.026 (1)
C(4′)	0.7867 (2)	0.6742 (5)	0.5800 (1)	0.023 (1)
C(5′)	0.8450 (2)	0.8231 (5)	0.5907 (1)	0.024 (1)
C(6′)	0.9476 (2)	0.8208 (5)	0.5925 (1)	0.027 (1)
C(7′)	0.9832 (2)	0.6693 (5)	0.5670 (1)	0.029 (1)
C(8′)	0.9275 (2)	0.5205 (5)	0.5597 (1)	0.030 (1)
C(9′)	1.0197 (2)	0.9586 (5)	0.6353 (1)	0.033 (1)
C(10′)	1.0746 (2)	0.9128 (5)	0.7264 (1)	0.032 (1)
C(11′)	1.0324 (2)	0.7528 (5)	0.7540 (1)	0.027 (1)
C(12′)	1.0709 (2)	0.5914 (5)	0.7448 (1)	0.031 (1)
C(13′)	1.0113 (2)	0.4463 (5)	0.7380 (1)	0.033 (1)
C(14′)	0.9107 (2)	0.4584 (5)	0.7398 (1)	0.032 (1)
C(15′)	0.8814 (2)	0.6134 (5)	0.7665 (1)	0.030 (1)
C(16′)	0.9410 (2)	0.7587 (5)	0.7741 (1)	0.028 (1)
C(17′)	0.6775 (2)	0.6894 (5)	0.5805 (1)	0.025 (1)
O(18′)	0.6154 (1)	0.5767 (5)	0.5454 (1)	0.036 (1)
O(19′)	0.6528 (1)	0.8201 (4)	0.6146 (1)	0.030 (1)
C(1″)	0.5626 (2)	0.1479 (5)	0.3634 (1)	0.024 (1)
C(2″)	0.5899 (2)	0.3338 (5)	0.3488 (1)	0.027 (1)
C(3″)	0.7024 (2)	0.3723 (5)	0.3790 (2)	0.035 (1)
C(4″)	0.6234 (2)	0.0221 (5)	0.3297 (1)	0.028 (1)
C(5″)	0.7102 (2)	−0.0556 (5)	0.3785 (2)	0.036 (1)
C(6″)	0.7646 (2)	−0.1701 (5)	0.3462 (2)	0.045 (1)
C(7″)	0.7333 (2)	−0.2098 (5)	0.2657 (2)	0.051 (1)
C(8″)	0.6470 (2)	−0.1332 (5)	0.2164 (2)	0.044 (1)
C(9″)	0.5925 (2)	−0.0171 (5)	0.2482 (1)	0.033 (1)

C(10'')	0.5105 (2)	0.6231 (5)	0.3554 (2)	0.039 (1)
N(11'')	0.5332 (1)	0.4484 (5)	0.3909 (1)	0.027 (1)
O(12'')	0.5789 (1)	0.1243 (5)	0.4473 (1)	0.035 (1)

The program system employed was Siemens *SHELXTL-Plus* (Sheldrick, 1990).

We thank the Fonds der Chemischen Industrie for financial support.

Lists of structure factors, anisotropic thermal parameters, H-atom coordinates, bond distances, bond angles and torsion angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55943 (42 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: HA1020]

References

Cram, D. J. & Allinger, N. L. (1955). *J. Am. Chem. Soc.* **77**, 6289–6294.
Cram, D. J. & Delton, M. H. (1970). *J. Am. Chem. Soc.* **92**, 7623–7625.
Cram, D. J. & Reich, H. J. (1969). *J. Am. Chem. Soc.* **91**, 3517–3526.
Falk, H., Reich-Rohrwig, P. & Schlögl, K. (1970). *Tetrahedron*, **26**, 511–527.
Laue, T. (1991). PhD thesis, Technical Univ. of Braunschweig, Germany.
Sheldrick, G. M. (1990). *SHELXTL-Plus*. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
Tochtermann, W., Olsson, G., Vogt, C., Peters, E.-M., Peters, K. & von Schnering, H. G. (1987). *Chem. Ber.* **120**, 1523–1532.
Vögtle, F. (1990). *Cyclophan-Chemie*, p. 91. Stuttgart: Teubner Studienbücher.

Acta Cryst. (1993). **C49**, 1195–1197

(−)-(*S*)-α-Phenylethylammonium (−)-(*R*)-[2.2]Paracyclophane-4-carboxylate

Peter G. Jones

Institut für Anorganische und Analytische Chemie der Technischen Universität, Hagenring 30, W-3300 Braunschweig, Germany

Thomas Laue and Henning Hopf

Institut für Organische Chemie der Technischen Universität, Hagenring 30, W-3300 Braunschweig, Germany

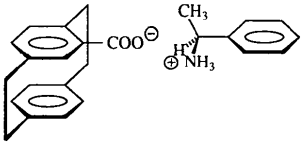
(*Received* 24 *June* 1992; *accepted* 6 *October* 1992)

Abstract

The anion, tricyclo[8.2.2.2^{4,7}]hexadeca-4,6,10,12,13,-15-hexaene-5-carboxylate, shows ring distortions typical of cyclophane systems. The cation forms three hydrogen bonds from the NH_3 group to carboxylate O atoms, with N···O distances 2.73–2.82 Å. The ions are thus linked to form hydrophilic ribbons parallel to the *y* axis.

Comment

As discussed in the previous paper (Jones, Döring, Laue & Hopf, 1993), substituted cyclophanes are chiral molecules, and we have therefore used (+)-(*S*)-[2.2]paracyclophane-4-carboxylic acid to resolve (±)-amines. However, it is first necessary to separate the enantiomers of the carboxylic acid, which can conveniently be achieved using (−)-(*S*)-α-phenylethylamine; full details of the separation are given by Laue (1991). Here we report the structure of the crystalline salt (−)-(*S*)-α-phenylethylammonium (−)-(*R*)-[2.2]paracyclophane-4-carboxylate (I), crystallized from ethanol.



(I)

Fig. 1 shows the asymmetric unit. Bond lengths and angles are normal. The cyclophane rings show the usual distortion (Vögtle, 1990), the bridgehead atoms lying 0.15–0.17 Å out of the plane of the remaining ring atoms. The carboxylate group is not coplanar with the ring [the dihedral angle between C(3), C(4), C(5) and C(17), O(1), O(2) is 45°].

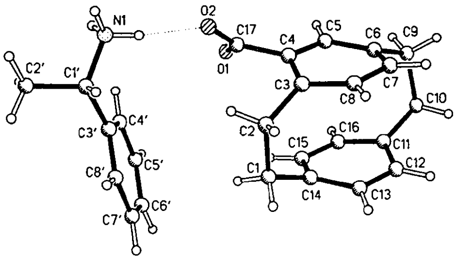


Fig. 1. The asymmetric unit of the title compound. Radii are arbitrary. The dotted line indicates a hydrogen bond.

H atoms were unambiguously located. Hydrogen bonds are formed from the cation —NH_3 to carboxylate O atoms [N(1)···O(2) 2.73, N(1)···O(2)($\frac{1}{2} - x$, $-\frac{1}{2} + y$, $1 - z$) 2.82, N(1)···O(1)($\frac{1}{2} - x$, $\frac{1}{2} + y$, $1 - z$) 2.74 Å]. The ions are thus linked to form a hydrophilic ribbon parallel to the short *y* axis (Fig. 2).

0108-2701/93/061195-03$06.00