

interactions with trenH_3^+ cations to one side of the chain of ions depicted in Fig. 1, but the third perchlorate anion in the sequence has no significant hydrogen-bonding interactions at all. It is of interest that there is no hydrogen bonding between trenH_3^+ cations, in contrast to the situation in, for example, $[\text{phen}_2\text{H}](\text{ClO}_4)$, where hydrogen bonds link pairs of 1,10-phenanthroline molecules (Maresca, Natile & Fanizzi, 1989).

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Structures of Two Crown-Ether Derivatives of 9-Acridone

BY A. VICHET, J. P. GALY, A. BALDY AND J. BARBE

URA CNRS 1411, *Faculté des Sciences de Saint Jérôme*,
Avenue Escadrille Normandie Niémen, 13397 Marseille CEDEX 13, France

AND J. FENEAU-DUPONT AND J.-P. DECLERCQ

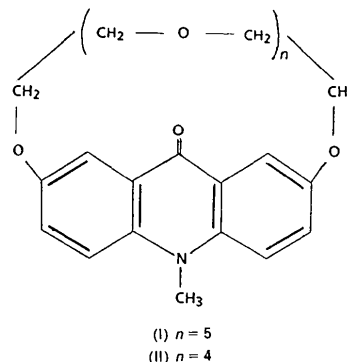
Laboratoire de chimie physique et de cristallographie, Université Catholique de Louvain,
1 place Louis Pasteur, 1348 Louvain la Neuve, Belgium

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Abstract. (I) *N*-Methyl-2,5,8,11,14,17,20-hepta-oxa-25-azatetracyclo[19.7.5.0^{24,32}.0^{26,30}]tritiaconta-1(29),-21(33),22,24(32),26(30),27-hexaen-31-one trihydrate, $\text{C}_{26}\text{H}_{33}\text{NO}_8 \cdot 3\text{H}_2\text{O}$, $M_r = 541.6$, triclinic, $P\bar{1}$, $a = 7.389$ (1), $b = 13.255$ (1), $c = 14.448$ (2) Å, $\alpha = 88.60$ (1), $\beta = 78.58$ (1), $\gamma = 81.48$ (1)°, $V = 1371.7$ (2) Å³, $Z = 2$, $D_x = 1.31$ g cm⁻³, $\lambda(\text{Cu } K\alpha) = 1.54178$ Å, $\mu = 8.68$ cm⁻¹, $F(000) = 580$, $T = 291$ K, $R = 0.047$ for 3376 observed reflections. (II) *N*-Methyl-2,5,8,11,14,17-hexa-oxa-22-azatetracyclo[16.7.5.0^{21,29}.0^{23,27}]triaconta-1(26),18(30),19,-21(29),23(27),24-hexaen-28-one, $\text{C}_{24}\text{H}_{29}\text{NO}_7$, $M_r = 443.5$, triclinic, $P\bar{1}$, $a = 8.426$ (2), $b = 11.420$ (2), $c = 11.739$ (4) Å, $\alpha = 87.20$ (3), $\beta = 79.33$ (3), $\gamma = 77.88$ (2)°, $V = 1085.3$ (5) Å³, $Z = 2$, $D_x = 1.36$ g cm⁻³, $\lambda(\text{Mo } K\alpha) = 0.71069$ Å, $\mu = 1.08$ cm⁻¹, $F(000) = 472$, $T = 291$ K, $R = 0.039$ for 3229 observed reflections. In these substituted acridone derivatives one molecule contains both a crown ether moiety with its complexing capabilities and a heterocyclic N atom able to modify these properties. In the two compounds, the crown ether chain is nearly perpendicular to the acridinic skeleton which is planar. In (I) the water molecules take part in hydrogen bonds with the crown ether [O37...O11 = 3.03 (1), O35...O36 = 2.81 (1) Å] and with each other [O36...O37 = 2.93 (1), O38...O36 = 2.84 (1), O38...O37 ($x + 1, y, z$) = 2.89 (1) and O38...O37 ($1 -$

$x, 1 - y, 1 - z$) = 2.88 (1) Å]. The bond lengths suggest partial localization of double bonds in the benzene rings.

Experimental. Crystals obtained by evaporation from ethanol. D_m not measured. Crystal sizes: (I) 0.31 × 0.22 × 0.13, (II) 0.2 × 0.2 × 0.4 mm. Lattice parameters refined using (I) 16, (II) 30 reflections in the



range (I) $6 \leq 2\theta \leq 38$, (II) $6 \leq 2\theta \leq 25$ °. Huber four-circle diffractometer, graphite-monochromated radiation, Cu $K\alpha$ for (I) and Mo $K\alpha$ for (II). For (I) 4936 $h \pm k \pm l$ independent reflections with $\sin\theta/\lambda \leq 0.60$ Å⁻¹, 3376 with $I \geq 2.5\sigma(I)$. For (II) 4269 $h \pm k$

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Table 1. Atomic coordinates ($\times 10^4$) and equivalent isotropic temperature factors ($\text{\AA}^2 \times 10^3$)
$$U_{\text{eq}} = (1/3) \sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i \cdot \mathbf{a}_j$$

Compound (I)	x	y	z	U_{eq}
C1	1549 (4)	8047 (2)	11269 (2)	48 (1)
O2	910 (3)	7523 (1)	12066 (1)	63 (1)
C3	826 (4)	6453 (2)	12011 (2)	56 (1)
C4	-989 (4)	6246 (2)	11798 (2)	54 (1)
O5	-929 (3)	6329 (1)	10817 (1)	52 (1)
C6	-2615 (4)	6172 (2)	10556 (2)	56 (1)
C7	-2273 (4)	6091 (2)	9500 (2)	60 (1)
O8	-1903 (3)	7038 (1)	9087 (1)	55 (1)
C9	-1205 (4)	6935 (2)	8106 (2)	57 (1)
C10	-800 (4)	7956 (2)	7732 (2)	50 (1)
O11	-198 (2)	7873 (1)	6739 (1)	52 (1)
C12	372 (4)	8785 (2)	6337 (2)	48 (1)
C13	806 (4)	8681 (2)	5284 (2)	51 (1)
O14	2241 (2)	7857 (1)	5005 (1)	49 (1)
C15	2486 (4)	7627 (2)	4027 (2)	53 (1)
C16	4322 (3)	6976 (2)	3711 (2)	49 (1)
O17	5794 (2)	7580 (1)	3625 (1)	44 (1)
C18	7167 (3)	7243 (2)	4168 (2)	49 (1)
C19	6492 (4)	7421 (2)	5210 (2)	46 (1)
O20	6111 (2)	8495 (1)	5379 (1)	44 (1)
C21	5366 (3)	8797 (2)	6293 (2)	37 (1)
C22	5045 (3)	9850 (2)	6455 (2)	45 (1)
C23	4353 (4)	10241 (2)	7343 (2)	46 (1)
C24	3890 (3)	9600 (2)	8117 (2)	37 (1)
N25	3232 (3)	9988 (1)	9021 (1)	41 (1)
C26	2696 (3)	9349 (2)	9761 (2)	40 (1)
C27	1968 (4)	9734 (2)	10685 (2)	51 (1)
C28	1429 (4)	9103 (2)	11409 (2)	53 (1)
C29	2244 (3)	7648 (2)	10379 (2)	41 (1)
C30	2836 (3)	8287 (2)	9627 (2)	36 (1)
C31	3586 (3)	7833 (2)	8695 (2)	36 (1)
C32	4133 (3)	8541 (2)	7945 (2)	34 (1)
C33	4888 (3)	8154 (2)	7027 (2)	36 (1)
C34	3094 (5)	11084 (2)	9192 (2)	58 (1)
O35	3729 (2)	6901 (1)	8559 (1)	46 (1)
O36	4443 (4)	5493 (2)	7062 (2)	76 (1)
O37	1523 (4)	5780 (2)	5935 (2)	76 (1)
O38	8079 (4)	4981 (2)	5952 (2)	82 (1)

Compound (II)	x	y	z	U_{eq}
C1	9787 (2)	8465 (1)	6900 (1)	47 (1)
O2	11459 (1)	8168 (1)	6414 (1)	58 (1)
C3	11949 (2)	8684 (2)	5287 (2)	56 (1)
C4	12850 (2)	9669 (2)	5342 (2)	55 (1)
O5	11738 (1)	10695 (1)	5851 (1)	56 (1)
C6	12488 (2)	11669 (2)	5967 (2)	57 (1)
C7	11219 (3)	12641 (2)	6608 (2)	60 (1)
O8	10127 (1)	13240 (1)	5877 (1)	55 (1)
C9	8774 (3)	14033 (2)	6524 (2)	60 (1)
C10	7673 (2)	14659 (2)	5712 (2)	60 (1)
O11	6144 (1)	15294 (1)	6343 (1)	61 (1)
C12	4975 (2)	14549 (2)	6688 (2)	61 (1)
C13	3536 (2)	15193 (2)	7537 (2)	58 (1)
O14	4013 (1)	15257 (1)	8627 (1)	53 (1)
C15	2652 (2)	15624 (2)	9551 (2)	57 (1)
C16	2268 (2)	14618 (2)	10344 (2)	54 (1)
O17	1497 (1)	13805 (1)	9849 (1)	57 (1)
C18	2398 (2)	12689 (1)	9506 (1)	43 (1)
C19	1483 (2)	11803 (2)	9487 (1)	48 (1)
C20	2236 (2)	10667 (2)	9122 (1)	47 (1)
C21	3957 (2)	10359 (1)	8750 (1)	39 (1)
N22	4732 (2)	9212 (1)	8359 (1)	42 (1)
C23	6404 (2)	8953 (1)	7892 (1)	40 (1)
C24	7187 (2)	7831 (2)	7391 (2)	52 (1)
C25	8831 (2)	7604 (2)	6912 (2)	54 (1)
C26	9067 (2)	9551 (1)	7397 (1)	46 (1)
C27	7383 (2)	9818 (1)	7889 (1)	41 (1)
C28	6662 (2)	11003 (1)	8384 (1)	44 (1)
C29	4878 (2)	11250 (1)	8768 (1)	39 (1)
C30	4072 (2)	12414 (1)	9151 (1)	43 (1)
C31	3765 (2)	8270 (2)	8481 (2)	56 (1)
O32	7515 (1)	11750 (1)	8462 (1)	64 (1)

Table 2. Bond distances (\AA)

(I)	(II)	(I)	(II)
O2—C1		1.369 (3)	1.394 (2)
C28—C1	C25—C1	1.407 (4)	1.394 (2)
C29—C1	C26—C1	1.373 (3)	1.368 (2)
C3—C2		1.433 (3)	1.446 (2)
C4—C3		1.499 (4)	1.492 (3)
O5—C4		1.411 (3)	1.417 (2)
C6—O5		1.415 (3)	1.413 (2)
C7—C6		1.500 (4)	1.492 (3)
O8—C7		1.418 (3)	1.422 (2)
C9—O8		1.411 (3)	1.417 (2)
C10—C9		1.494 (4)	1.504 (3)
O11—C10		1.418 (3)	1.429 (2)
C12—O11		1.413 (3)	1.425 (2)
C13—C12		1.496 (4)	1.501 (3)
O14—C13		1.410 (3)	1.419 (2)
C15—O14		1.424 (3)	1.432 (2)
C16—C15		1.489 (4)	1.494 (3)
O17—C16		1.428 (3)	1.438 (2)
C18—O17	—	1.417 (3)	—
C19—C18	—	1.502 (3)	—
O20—C19	—	1.426 (3)	—
C21—O20	C18—O17	1.370 (3)	1.375 (2)
C22—C21	C19—C18	1.399 (3)	1.399 (2)
C33—C21	C30—C18	1.370 (3)	1.368 (2)
C23—C22	C20—C19	1.368 (3)	1.370 (2)
C24—C23	C21—C20	1.406 (3)	1.408 (2)
N25—C24	N22—C21	1.383 (3)	1.394 (2)
C32—C24	C29—C21	1.411 (3)	1.407 (2)
C26—N25	C23—N22	1.380 (3)	1.388 (2)
C34—N25	C31—N22	1.465 (3)	1.467 (2)
C27—C26	C24—C23	1.414 (3)	1.413 (2)
C30—C26	C27—C23	1.411 (3)	1.414 (2)
C28—C27	C25—C24	1.358 (4)	1.370 (2)
C30—C29	C27—C26	1.403 (3)	1.404 (2)
C31—C30	C28—C27	1.459 (3)	1.463 (2)
C32—C31	C29—C28	1.451 (3)	1.458 (2)
O35—C31	O32—C28	1.241 (2)	1.241 (2)
C33—C32	C30—C29	1.410 (3)	1.412 (2)

deviation. Structure solved by direct methods using *SHELXS86* (Sheldrick, 1985). H atoms for (I) in computed positions, except those in the methyl group (C34) and on the water molecules, from difference Fourier synthesis; for (II) from difference Fourier synthesis. Anisotropic least-squares refinement (*SHELX76*; Sheldrick, 1976) using *F*; H atoms isotropic with common refined temperature factor ($U = 0.07 \text{\AA}^2$), $w = 1/(\sigma^2 + gF^2)$; $g = 0.004$ for (I) and 0.00002 for (II). (I) $R = 0.047$, $wR = 0.060$, $S = 1.04$ for 3376 observed reflections; (II) $R = 0.039$, $wR = 0.038$, $S = 2.5$ for 3229 observed reflections. Final max. shift to e.s.d. = 0.3 (I), 0.06 (II). Max. and min. heights in final difference Fourier synthesis for (I) 0.29 and -0.24 , and for (II) 0.20 and -0.13 e \AA^{-3} . Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV). The atomic parameters are given in Table 1,* the bond distances in Table 2 and the bond angles in Table 3. Figs. 1 and 2 give stereoscopic views of the molecules, showing the numbering of the atoms.

* Lists of structure factors, anisotropic thermal parameters, bond lengths and angles including H atoms and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54358 (44 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

± 1 independent reflections with $\sin \theta / \lambda \leq 0.62 \text{\AA}^{-1}$, 3229 with $I \geq 2.5\sigma(I)$. Standard reflections (I) 223, (II) 352, checked every 50 reflections; no significant

Table 3. Bond angles (°)

(I)	(II)	(I)	(II)
C28—C1—O2	C25—C1—O2	114.2 (2)	118.5 (1)
C29—C1—O2	C26—C1—O2	126.9 (2)	122.5 (1)
C29—C1—C28	C26—C1—C25	119.0 (2)	118.9 (2)
C3—O2—C1		120.1 (2)	116.5 (1)
C4—C3—O2		112.4 (2)	112.6 (1)
O5—C4—C3		109.5 (2)	109.9 (1)
C6—O5—C4		113.2 (2)	114.1 (1)
C7—C6—O5		108.1 (2)	108.5 (1)
O8—C7—C6		110.1 (2)	110.7 (1)
C9—O8—C7		111.3 (2)	110.9 (1)
C10—C9—O8		107.9 (2)	108.8 (1)
O11—C10—C9		108.4 (2)	110.8 (1)
C12—O11—C10		112.0 (2)	112.6 (1)
C13—C12—O11		109.2 (2)	110.1 (1)
O14—C13—C12		110.2 (2)	109.8 (2)
C15—O14—C13		111.9 (2)	113.6 (1)
C16—C15—O14		109.5 (2)	113.2 (1)
O17—C16—C15		109.9 (2)	113.8 (2)
C18—O17—C16		115.1 (2)	—
C19—C18—O17		113.6 (2)	—
O20—C19—C18		108.4 (2)	—
C21—O20—C19	C18—O17—C16	116.2 (2)	119.4 (1)
C22—C21—O20	C19—C18—O17	115.7 (2)	115.7 (1)
C33—C21—O20	C30—C18—O17	125.0 (2)	125.0 (1)
C33—C21—C22	C30—C18—C19	119.3 (2)	119.2 (1)
C23—C22—C21	C20—C19—C18	120.9 (2)	121.1 (1)
C24—C23—C22	C21—C20—C19	121.2 (2)	120.9 (1)
N25—C24—C23	N22—C21—C20	121.5 (2)	121.3 (1)
C32—C24—C23	C29—C21—C20	117.8 (2)	118.1 (1)
C32—C24—N25	C29—C21—N22	120.7 (2)	120.6 (1)
C26—N25—C24	C23—N22—C21	120.4 (2)	120.7 (1)
C34—N25—C24	C31—N22—C21	119.8 (2)	118.9 (1)
C34—N25—C26	C31—N22—C23	119.9 (2)	120.3 (1)
C27—C26—N25	C24—C23—N22	121.2 (2)	121.8 (1)
C30—C26—N25	C27—C23—N22	121.5 (2)	120.7 (1)
C30—C26—C27	C27—C23—C24	117.3 (2)	117.5 (1)
C28—C27—C26	C25—C24—C23	121.0 (2)	120.8 (2)
C27—C28—C1	C24—C25—C1	121.5 (2)	121.5 (2)
C30—C29—C1	C27—C26—C1	120.3 (2)	121.2 (1)
C29—C30—C26	C26—C27—C23	121.0 (2)	120.1 (1)
C31—C30—C26	C28—C27—C23	120.4 (2)	120.8 (1)
C31—C30—C29	C28—C27—C26	118.6 (2)	119.1 (1)
C32—C31—C30	C29—C28—C27	115.7 (2)	115.5 (1)
O35—C31—C30	O32—C28—C27	121.6 (2)	122.0 (1)
O35—C31—C32	O32—C28—C29	122.7 (2)	122.4 (1)
C31—C32—C24	C28—C29—C21	121.3 (2)	121.2 (1)
C33—C32—C24	C30—C29—C21	120.0 (2)	120.0 (1)
C33—C32—C31	C30—C29—C28	118.8 (2)	118.8 (1)
C32—C33—C21	C29—C30—C18	120.7 (2)	120.8 (1)

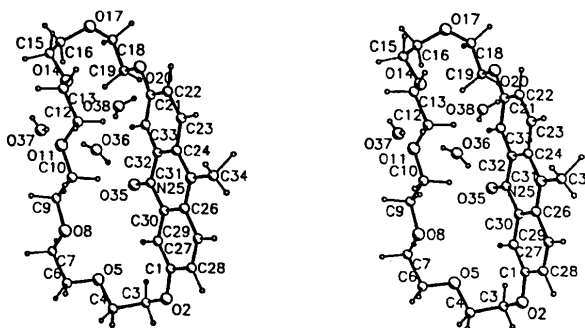


Fig. 1. Stereoscopic view and atom numbering of (I).

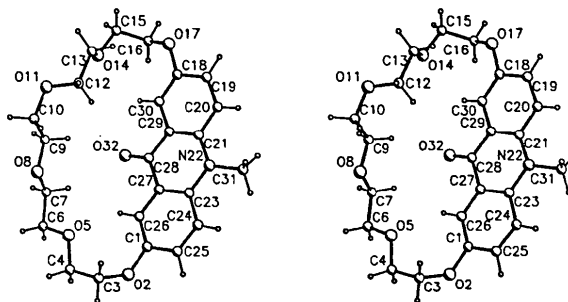


Fig. 2. Stereoscopic view and atom numbering of (II).

1(25),23-diene-2,22-dione benzylammonium perchlorate (Dalley, Bradshaw, Larson & Simonsen, 1982).

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Related literature. As far as we know, these studies are the first descriptions of the structure of crown-ether derivatives of acridine. The geometry of the acridone moieties closely resembles 9-[3-(methylamino)propyl]imino-1-nitro-9,10-dihydroacridine (Stezowski, Kollat, Bogucka-Ledochowska & Glusker, 1985), *N*-methylacridone (Dzyabchenko, Zavodnik & Belbskii, 1980), *N*-ethylacridone (Zavodnik, Chetkina & Valbkova, 1979) and *N*-phenylacridone (Zavodnik, Chetkina & Valbkova, 1981). In all of these, partial double bonds in benzo rings are observed. The structures of a few polycyclic molecules involving $-(O-CH_2-CH_2)_6-O-$ and $-(O-CH_2-CH_2)_7-O-$ chains have been described: (1*R*,2*R*,7*R*,24*R*)-3,5,8,11,14,17,20,23-, 26,28-decaoxatricyclo[21.4.0.0^{2,7}]octacosaneborane-ammonia clathrate (Shahriari-Zavareh, Stoddart, Williams, Allwood & Williams, 1985) and 3,6,9,12,15,18,21,26-octaoxabicyclo[21.2.1]hexacosa-