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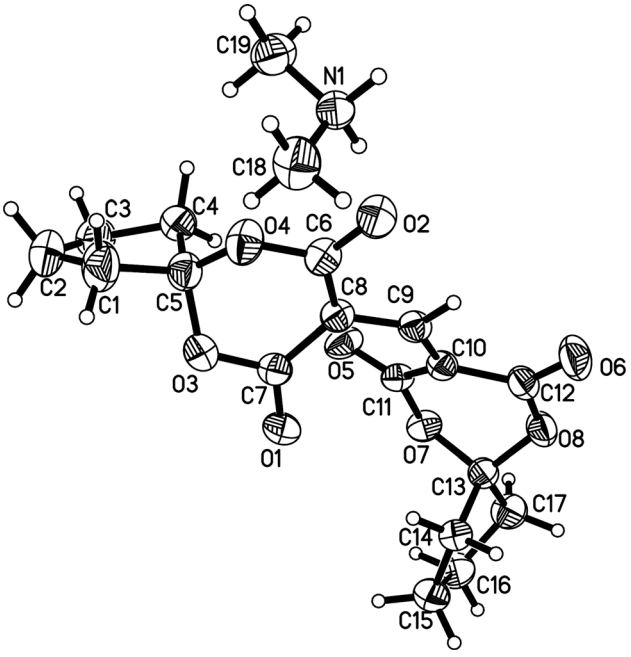
# The crystal structure of dimethylammonium 8-[(7,9-dioxo-6,10-dioxaspiro[4.5]decan-8-ylidene)methyl]-9-oxo-6,10-dioxaspiro[4.5]dec-7-en-7-olate, $C_{19}H_{25}NO_8$

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**Abstract**

$C_{19}H_{25}NO_8$, monoclinic, $P2_1/n$ (no. 14), $a$ = 10.149(2) Å, $b$ = 12.338(3) Å, $c$ = 15.898(3) Å, $\beta$ = 97.58(3)°, $V$ = 1973.2(7) Å$^3$, $Z$ = 4, $R_{gt}(F)$ = 0.0581, $wR_{ref}(F^2)$ = 0.1842, T = 293(2) K.

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**Table 1:** Data collection and handling.

| | |
|---|---|
| Crystal: | Yellow block |
| Size: | 0.20 × 0.16 × 0.12 mm |
| Wavelength: | Mo $K\alpha$ radiation (0.71073 Å) |
| $\mu$: | 0.10 mm$^{-1}$ |
| Diffractometer, scan mode: | Rigaku Spider Rapid IP, $\omega$ |
| $\theta_{max}$, completeness: | 27.5°, >99% |
| $N(hkl)_{measured}$, $N(hkl)_{unique}$, $R_{int}$: | 18636, 4510, 0.028 |
| Criterion for $I_{obs}$, $N(hkl)_{gt}$: | $I_{obs} > 2\ \sigma(I_{obs})$, 3509 |
| $N(param)_{refined}$: | 253 |
| Programs: | RAPID [1], SHELX [2, 3] |

The molecular structure is shown in the figure. Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

## Source of material

IR spectra were performed on FT IR-650 instrument. The starting material 6,10-dioxaspiro[4.5]decane-7,9-dione (1.84, 0.01 mol), dissolved in absolute ethanol (25 mL) was added to a round-bottom flask and magnetically stirred at 25 °C. Then, 1,1-dimethoxy-*N*,*N*-dimethylmethanamine (1.19 g, 0.01 mol) was added dropwise to the mixture for half an hour. The reaction mixture was kept stirring for 2.5 h, then 1,1-dimethoxy-*N*,*N*-dimethylmethan amine (0.119 g, 0.001 mol) was added again. The above mixture was set aside for another 2 h. Then the solution is cooled and evaporated at room temperature. The precipitate was collected by filtration, washed three times and dried. Yield 27.6%. M.p.: 137.8–138.1 °C. The colorless block-shaped crystals of $C_{19}H_{25}NO_8$ were obtained by evaporation of a solution ($V_{petroleumether}$:$V_{acetone}$ = 2:1). Peaks at 1717, 1672, 1253, 1147 cm$^{-1}$ are attributed to the stretching vibrations of C=O and C–O bands of 1,3-dioxane ring.

## Experimental details

The structure was solved by SHELXT-2015 [2] and refined with SHELXL-2015 [3]. The value of $U_{iso}$(H) is 1.2 times $U_{eq}$ of

**Table 2:** Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å$^2$).

| Atom | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| C1 | 0.0582 (2) | 0.7744 (3) | 0.16269 (19) | 0.1016 (8) |
| H1A | 0.016646 | 0.707336 | 0.141354 | 0.122* |
| H1B | 0.010620 | 0.802198 | 0.207187 | 0.122* |
| C2 | 0.0573 (3) | 0.8549 (2) | 0.0933 (2) | 0.1230 (11) |
| H2A | 0.028479 | 0.820712 | 0.039043 | 0.148* |
| H2B | −0.003830 | 0.913301 | 0.101207 | 0.148* |
| C3 | 0.1929 (3) | 0.8981 (2) | 0.09462 (15) | 0.1006 (8) |
| H3A | 0.191210 | 0.976620 | 0.091608 | 0.121* |
| H3B | 0.233136 | 0.870410 | 0.046872 | 0.121* |
| C4 | 0.2712 (2) | 0.86054 (15) | 0.17823 (12) | 0.0706 (5) |
| H4A | 0.364026 | 0.848653 | 0.172219 | 0.085* |
| H4B | 0.265458 | 0.913094 | 0.222977 | 0.085* |
| C5 | 0.20377 (18) | 0.75584 (16) | 0.19627 (12) | 0.0673 (4) |
| C6 | 0.33382 (17) | 0.68909 (14) | 0.32116 (10) | 0.0603 (4) |
| C7 | 0.37644 (18) | 0.62979 (13) | 0.17834 (10) | 0.0607 (4) |
| C8 | 0.42601 (16) | 0.65072 (12) | 0.26652 (9) | 0.0537 (4) |
| C9 | 0.54908 (16) | 0.61385 (12) | 0.30560 (9) | 0.0532 (4) |
| H9 | 0.555255 | 0.605403 | 0.364119 | 0.064* |
| C10 | 0.66246 (16) | 0.58808 (12) | 0.27147 (9) | 0.0521 (3) |
| C11 | 0.69319 (16) | 0.63315 (11) | 0.19257 (9) | 0.0523 (3) |
| C12 | 0.76714 (18) | 0.52760 (15) | 0.32288 (10) | 0.0634 (4) |
| C13 | 0.83700 (16) | 0.48106 (13) | 0.19135 (10) | 0.0567 (4) |
| C14 | 0.73605 (18) | 0.39344 (13) | 0.16208 (10) | 0.0608 (4) |
| H14A | 0.646568 | 0.422735 | 0.154736 | 0.073* |
| H14B | 0.740876 | 0.334662 | 0.202886 | 0.073* |
| C15 | 0.7741 (2) | 0.35405 (17) | 0.07757 (12) | 0.0766 (5) |
| H15A | 0.782773 | 0.275755 | 0.077907 | 0.092* |
| H15B | 0.706294 | 0.374459 | 0.031480 | 0.092* |
| C16 | 0.9053 (2) | 0.4065 (2) | 0.06607 (14) | 0.0832 (6) |
| H16A | 0.892135 | 0.464701 | 0.024814 | 0.100* |
| H16B | 0.965695 | 0.353697 | 0.046959 | 0.100* |
| C17 | 0.96002 (19) | 0.45015 (19) | 0.15271 (14) | 0.0777 (5) |
| H17A | 1.010821 | 0.395182 | 0.186477 | 0.093* |
| H17B | 1.016304 | 0.512755 | 0.147783 | 0.093* |
| C18 | 0.0416 (2) | 0.6622 (2) | 0.46716 (17) | 0.0914 (6) |
| H18A | 0.068424 | 0.592019 | 0.489224 | 0.137* |
| H18B | −0.036059 | 0.685346 | 0.490754 | 0.137* |
| H18C | 0.021790 | 0.658278 | 0.406501 | 0.137* |
| C19 | 0.1189 (2) | 0.85049 (17) | 0.45933 (14) | 0.0816 (6) |
| H19A | 0.193778 | 0.896636 | 0.476634 | 0.122* |
| H19B | 0.100555 | 0.850048 | 0.398523 | 0.122* |
| H19C | 0.042706 | 0.877115 | 0.482775 | 0.122* |
| N1 | 0.14923 (14) | 0.73976 (12) | 0.49000 (8) | 0.0606 (4) |
| H1C | 0.221315 | 0.716586 | 0.469052 | 0.073* |
| H1D | 0.168302 | 0.741391 | 0.546262 | 0.073* |
| O1 | 0.42965 (15) | 0.57573 (12) | 0.12902 (8) | 0.0790 (4) |
| O2 | 0.35362 (14) | 0.68833 (12) | 0.39906 (7) | 0.0756 (4) |
| O3 | 0.25108 (13) | 0.66764 (11) | 0.15063 (8) | 0.0734 (4) |
| O4 | 0.21593 (13) | 0.72885 (12) | 0.28451 (9) | 0.0760 (4) |
| O5 | 0.64653 (14) | 0.71522 (9) | 0.15935 (8) | 0.0704 (4) |
| O6 | 0.77449 (17) | 0.51038 (15) | 0.39792 (8) | 0.0931 (5) |
| O7 | 0.79328 (11) | 0.58501 (9) | 0.15707 (7) | 0.0595 (3) |
| O8 | 0.86736 (12) | 0.49047 (11) | 0.28114 (7) | 0.0714 (4) |

all C(H) groups, all C(H,H) groups, all N(H,H) groups and 1.5 times of all C(H,H,H) groups. The secondary C(H, H) and methylic H are both refined with riding coordinates.

## Comment

New spiro compounds and their derivatives have been extensively studied in the biological field such as cytotoxicity [5], antimycobacterial [6], anti-inflammatory [7], cytotoxic activity [8, 9], antifungal activity [10], antitumor activity [11], antioxidants and anti-cancer agents [12]. Furthermore, application of spiro compounds in other aspects has also drawn great attention such as green corrosion inhibitors for mild steel [13], organic semiconductor [14] and emitting materials [15]. Based on above reasons, a lot of spiro compounds were prepared by our group for 10 years [16–19]. As part of our ongoing research, a new salt was received.

The title compound is a salt containing one $C_{17}H_{17}O_8$ anion and one $C_2H_8N$ cation. The two 6,10-dioxaspiro groups are linked to the central C(9) atom, which forms a conjugated system (C(8)=C(9)–C(10)). From the bond length data, the bond lengths of C8–C9 (1.396(2) Å), C9–C10 (1.373(2) Å) are same as that of our earlier report C(10)–C(11) (1.396(2) Å), C(8)–C(10) (1.373(2) Å) [4]. The bond angle of C(8)–C(9)–C(10) 130.33(14)° is consistent with that of C(8)–C(10)–C(12) 131.80(16)° [4]. The two cyclopentane rings of the $C_{17}H_{17}O_8$ anion both show half chair conformations. However, two 1,3-dioxane-4,6-dione rings both show envelope conformations.

There exists one kind of N(1)–H(1C)···O(2) intramolecular hydrogen bonds and one kind of N(1)–H(1D)···O(5) intermolecular hydrogen bonds in the title compound. The distance of N(1)···O(2) (2.754(19) Å) is in agreement to what was previously reported (2.750(2)°) [4]. The distance of N(1)···O(5) is 2.753(19) Å), symmetry code: $-1/2 + x$, $3/2 - y$, $1/2 + z$.

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**Conflict of interest statement:** The authors declare no conflicts of interest regarding this article.

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