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# Bis(carboxymethyl)ammonium 4-toluenesulfonate

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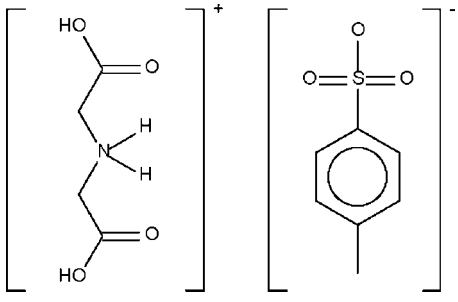
Key indicators: single-crystal X-ray study; $T$ = 100 K; mean $\sigma$(C–C) = 0.003 Å; $R$ factor = 0.038; $wR$ factor = 0.119; data-to-parameter ratio = 15.4.

The iminodiacetic acid component of the title salt, $C_4H_8NO_4^+\cdot C_7H_7SO_3^-$, is protonated at the N atom. The cation uses the ammonium group to form hydrogen bonds to the O atoms of two adjacent sulfonate groups. In addition, the carboxylic acid portions of the cation form hydrogen bonds to the sulfonate groups. The hydrogen-bonding interactions give rise to a layer structure.

## Related literature

For the crystal structures of iminodiacetic acid hydrohalides, see: Oskarsson (1973, 1974*a*,*b*, 1976).



## Experimental

### Crystal data

$C_4H_8NO_4^+\cdot C_7H_7O_3S^-$
$M_r$ = 305.30
Orthorhombic, *Pbca*
$a$ = 9.9291 (2) Å
$b$ = 10.3636 (2) Å
$c$ = 25.8862 (5) Å
$V$ = 2663.72 (9) Å$^3$
$Z$ = 8
Mo $K\alpha$ radiation
$\mu$ = 0.28 mm$^{-1}$
$T$ = 100 (2) K
0.27 × 0.27 × 0.27 mm

### Data collection

Bruker SMART APEX diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min}$ = 0.929, $T_{max}$ = 0.929
20842 measured reflections
3059 independent reflections
2560 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.048

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.038
$wR(F^2)$ = 0.119
$S$ = 1.15
3059 reflections
198 parameters
4 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.42 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.50 e Å$^{-3}$

**Table 1**
Hydrogen-bond geometry (Å, °).

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| N1—H1*N*1···O2$^{i}$ | 0.88 (1) | 2.02 (1) | 2.885 (2) | 167 (2) |
| N1—H1*N*2···O3$^{ii}$ | 0.88 (1) | 2.06 (2) | 2.792 (2) | 140 (2) |
| O5—H5*O*···O1 | 0.84 (1) | 1.79 (1) | 2.607 (2) | 164 (3) |
| O7—H7*O*···O2$^{iii}$ | 0.84 (1) | 1.85 (1) | 2.659 (2) | 160 (3) |

Symmetry codes: (i) $-x+1, y-\frac{1}{2}, -z+\frac{1}{2}$; (ii) $-x+\frac{1}{2}, y-\frac{1}{2}, z$; (iii) $x-\frac{1}{2}, y-1, -z+\frac{1}{2}$.

Data collection: *APEX2* (Bruker, 2007); cell refinement: *SAINT* (Bruker, 2007); data reduction: *SAINT*); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *X-SEED* (Barbour, 2001); software used to prepare material for publication: *publCIF* (Westrip, 2008).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BH2187).

## References

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# supporting information

*Acta Cryst.* (2008). E**64**, o1798 [doi:10.1107/S1600536808026214]

## Bis(carboxymethyl)ammonium 4-toluenesulfonate

**Kong Mun Lo and Seik Weng Ng**

### S1. Experimental

Iminodiacetic acid (0.55 g, 4 mmol) and *p*-toluenesulfonic acid (0.65 g, 4 mmol) were heated in toluene (100 ml) for 1 h. Crystals were isolated from the cool solution after several days.

### S2. Refinement

Carbon-bound H-atoms were placed in calculated positions (C—H 0.95 to 0.99 Å) and were included in the refinement in the riding model approximation, with $U_{iso}$(H) set to 1.2 to 1.5$U_{eq}$(carrier C). The acid and ammonium H atoms were refined with distance restraints of O—H = 0.84 (1) and N—H = 0.88 (1) Å; their isotropic displacement parameters were freely refined.

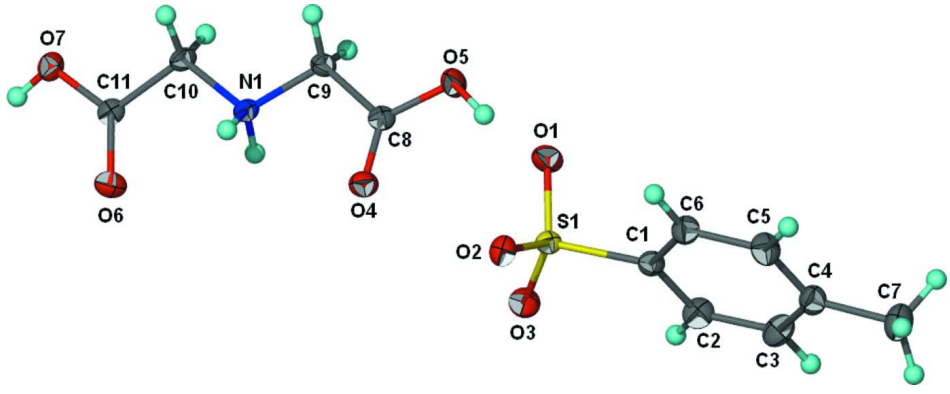


**Figure 1**

Thermal ellipsoid plot (Barbour, 2001) of $(C_4H_8NO_4)^+(C_7H_7O_3S)^-$ at the 70% probability level. Hydrogen atoms are drawn as spheres of arbitrary radius.

### Bis(carboxymethyl)ammonium 4-toluenesulfonate

*Crystal data*

$C_4H_8NO_4^+ \cdot C_7H_7O_3S^-$
$M_r$ = 305.30
Orthorhombic, *Pbca*
Hall symbol: -P 2ac 2ab
*a* = 9.9291 (2) Å
*b* = 10.3636 (2) Å
*c* = 25.8862 (5) Å
*V* = 2663.72 (9) Å$^3$
*Z* = 8
*F*(000) = 1280
$D_x$ = 1.523 Mg m$^{-3}$
Mo *Kα* radiation, *λ* = 0.71073 Å
Cell parameters from 4293 reflections
*θ* = 2.9–27.2°
*μ* = 0.28 mm$^{-1}$
*T* = 100 K
Triangular block, colorless
0.27 × 0.27 × 0.27 mm

*Data collection*

Bruker SMART APEX diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
$\omega$ scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min}$ = 0.930, $T_{max}$ = 0.930
20842 measured reflections
3059 independent reflections
2560 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.048
$\theta_{max}$ = 27.5°, $\theta_{min}$ = 1.6°
$h$ = −12→7
$k$ = −13→13
$l$ = −33→33

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.038
$wR(F^2)$ = 0.119
$S$ = 1.15
3059 reflections
198 parameters
4 restraints
0 constraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0644P)^2 + 0.8206P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.42 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.50 e Å$^{-3}$

*Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)*

| | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| S1 | 0.49041 (5) | 0.84948 (4) | 0.358654 (17) | 0.01248 (14) |
| O1 | 0.55419 (15) | 0.74172 (13) | 0.38508 (5) | 0.0195 (3) |
| O2 | 0.56209 (14) | 0.88382 (13) | 0.31102 (5) | 0.0163 (3) |
| O3 | 0.34727 (14) | 0.83294 (13) | 0.35029 (5) | 0.0190 (3) |
| O4 | 0.40223 (14) | 0.52637 (13) | 0.30853 (5) | 0.0171 (3) |
| O5 | 0.51261 (16) | 0.49339 (13) | 0.38301 (5) | 0.0199 (3) |
| H5O | 0.510 (3) | 0.5747 (10) | 0.3841 (10) | 0.034 (7)* |
| O6 | 0.20627 (14) | 0.17528 (13) | 0.21695 (5) | 0.0176 (3) |
| O7 | 0.29656 (14) | −0.02406 (13) | 0.22217 (5) | 0.0163 (3) |
| H7O | 0.229 (2) | −0.047 (3) | 0.2048 (10) | 0.051 (9)* |
| N1 | 0.37466 (17) | 0.27383 (15) | 0.28845 (6) | 0.0129 (3) |
| H1N1 | 0.402 (3) | 0.316 (2) | 0.2610 (7) | 0.031 (7)* |
| H1N2 | 0.2899 (11) | 0.296 (2) | 0.2924 (8) | 0.014 (5)* |
| C1 | 0.50864 (19) | 0.98391 (18) | 0.40013 (7) | 0.0141 (4) |
| C2 | 0.3995 (2) | 1.02980 (19) | 0.42797 (8) | 0.0187 (4) |
| H2 | 0.3146 | 0.9879 | 0.4256 | 0.022* |
| C3 | 0.4152 (2) | 1.1376 (2) | 0.45941 (8) | 0.0212 (4) |
| H3 | 0.3404 | 1.1681 | 0.4788 | 0.025* |
| C4 | 0.5374 (2) | 1.20164 (19) | 0.46306 (7) | 0.0183 (4) |
| C5 | 0.6471 (2) | 1.15329 (19) | 0.43556 (8) | 0.0191 (4) |
| H5 | 0.7319 | 1.1952 | 0.4380 | 0.023* |
| C6 | 0.6335 (2) | 1.04440 (19) | 0.40462 (7) | 0.0171 (4) |
| H6 | 0.7093 | 1.0112 | 0.3866 | 0.021* |
| C7 | 0.5517 (2) | 1.3217 (2) | 0.49545 (8) | 0.0247 (5) |
| H7A | 0.4636 | 1.3631 | 0.4993 | 0.037* |

| | | | | |
|---|---|---|---|---|
| H7B | 0.6142 | 1.3815 | 0.4785 | 0.037* |
| H7C | 0.5868 | 1.2985 | 0.5296 | 0.037* |
| C8 | 0.45172 (19) | 0.45546 (18) | 0.34029 (7) | 0.0137 (4) |
| C9 | 0.45326 (19) | 0.31071 (18) | 0.33494 (7) | 0.0137 (4) |
| H9A | 0.5472 | 0.2798 | 0.3315 | 0.016* |
| H9B | 0.4132 | 0.2705 | 0.3660 | 0.016* |
| C10 | 0.3811 (2) | 0.13288 (17) | 0.27813 (7) | 0.0142 (4) |
| H10A | 0.3570 | 0.0845 | 0.3098 | 0.017* |
| H10B | 0.4738 | 0.1085 | 0.2680 | 0.017* |
| C11 | 0.28461 (19) | 0.09925 (18) | 0.23534 (7) | 0.0132 (4) |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| S1 | 0.0104 (2) | 0.0118 (2) | 0.0153 (2) | −0.00084 (17) | −0.00072 (16) | 0.00057 (16) |
| O1 | 0.0240 (8) | 0.0125 (7) | 0.0221 (7) | −0.0013 (6) | −0.0072 (6) | 0.0031 (5) |
| O2 | 0.0164 (7) | 0.0158 (7) | 0.0168 (7) | −0.0021 (6) | 0.0028 (5) | −0.0016 (5) |
| O3 | 0.0109 (7) | 0.0226 (8) | 0.0233 (7) | −0.0016 (6) | −0.0006 (6) | −0.0024 (5) |
| O4 | 0.0169 (7) | 0.0148 (7) | 0.0197 (7) | 0.0007 (6) | −0.0025 (5) | 0.0007 (5) |
| O5 | 0.0293 (9) | 0.0111 (7) | 0.0192 (7) | −0.0007 (6) | −0.0092 (6) | −0.0014 (5) |
| O6 | 0.0156 (7) | 0.0155 (7) | 0.0217 (7) | −0.0016 (6) | −0.0030 (5) | 0.0033 (5) |
| O7 | 0.0134 (7) | 0.0143 (7) | 0.0211 (7) | −0.0023 (6) | −0.0006 (6) | −0.0043 (5) |
| N1 | 0.0123 (8) | 0.0109 (8) | 0.0154 (8) | 0.0011 (6) | −0.0002 (6) | 0.0000 (6) |
| C1 | 0.0144 (9) | 0.0131 (9) | 0.0149 (8) | −0.0001 (7) | −0.0003 (7) | 0.0010 (7) |
| C2 | 0.0148 (10) | 0.0198 (10) | 0.0215 (9) | −0.0019 (8) | 0.0032 (8) | 0.0004 (8) |
| C3 | 0.0190 (11) | 0.0217 (10) | 0.0228 (10) | 0.0025 (8) | 0.0066 (8) | −0.0020 (8) |
| C4 | 0.0237 (11) | 0.0162 (10) | 0.0150 (9) | 0.0007 (8) | −0.0001 (8) | 0.0004 (7) |
| C5 | 0.0145 (10) | 0.0225 (10) | 0.0202 (9) | −0.0045 (8) | 0.0002 (8) | −0.0023 (8) |
| C6 | 0.0130 (9) | 0.0199 (10) | 0.0183 (9) | 0.0004 (8) | 0.0014 (7) | −0.0027 (7) |
| C7 | 0.0276 (12) | 0.0219 (11) | 0.0247 (10) | −0.0011 (9) | 0.0010 (9) | −0.0067 (8) |
| C8 | 0.0102 (9) | 0.0150 (9) | 0.0161 (8) | −0.0005 (7) | 0.0005 (7) | 0.0004 (7) |
| C9 | 0.0125 (9) | 0.0133 (9) | 0.0152 (9) | 0.0002 (7) | −0.0026 (7) | 0.0000 (7) |
| C10 | 0.0132 (9) | 0.0095 (8) | 0.0200 (9) | 0.0004 (7) | −0.0017 (7) | −0.0009 (7) |
| C11 | 0.0108 (9) | 0.0132 (9) | 0.0157 (8) | −0.0019 (7) | 0.0028 (7) | 0.0012 (7) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| S1—O3 | 1.4478 (14) | C2—H2 | 0.9500 |
| S1—O1 | 1.4547 (14) | C3—C4 | 1.386 (3) |
| S1—O2 | 1.4675 (13) | C3—H3 | 0.9500 |
| S1—C1 | 1.768 (2) | C4—C5 | 1.394 (3) |
| O4—C8 | 1.207 (2) | C4—C7 | 1.507 (3) |
| O5—C8 | 1.320 (2) | C5—C6 | 1.390 (3) |
| O5—H5o | 0.84 (1) | C5—H5 | 0.9500 |
| O6—C11 | 1.205 (2) | C6—H6 | 0.9500 |
| O7—C11 | 1.328 (2) | C7—H7A | 0.9800 |
| O7—H7o | 0.84 (1) | C7—H7B | 0.9800 |
| N1—C9 | 1.484 (2) | C7—H7C | 0.9800 |

| | | | |
|---|---|---|---|
| N1—C10 | 1.486 (2) | C8—C9 | 1.507 (3) |
| N1—H1n1 | 0.88 (1) | C9—H9A | 0.9900 |
| N1—H1n2 | 0.88 (1) | C9—H9B | 0.9900 |
| C1—C2 | 1.385 (3) | C10—C11 | 1.505 (3) |
| C1—C6 | 1.394 (3) | C10—H10A | 0.9900 |
| C2—C3 | 1.391 (3) | C10—H10B | 0.9900 |

| | | | |
|---|---|---|---|
| O3—S1—O1 | 114.00 (9) | C4—C5—H5 | 119.7 |
| O3—S1—O2 | 112.28 (8) | C5—C6—C1 | 119.93 (18) |
| O1—S1—O2 | 111.72 (8) | C5—C6—H6 | 120.0 |
| O3—S1—C1 | 106.53 (9) | C1—C6—H6 | 120.0 |
| O1—S1—C1 | 105.95 (9) | C4—C7—H7A | 109.5 |
| O2—S1—C1 | 105.63 (8) | C4—C7—H7B | 109.5 |
| C8—O5—H5O | 108.2 (18) | H7A—C7—H7B | 109.5 |
| C11—O7—H7O | 110 (2) | C4—C7—H7C | 109.5 |
| C9—N1—C10 | 112.10 (14) | H7A—C7—H7C | 109.5 |
| C9—N1—H1N1 | 111.7 (17) | H7B—C7—H7C | 109.5 |
| C10—N1—H1N1 | 109.4 (16) | O4—C8—O5 | 125.13 (18) |
| C9—N1—H1N2 | 110.2 (14) | O4—C8—C9 | 123.20 (17) |
| C10—N1—H1N2 | 108.3 (15) | O5—C8—C9 | 111.66 (15) |
| H1N1—N1—H1N2 | 105 (2) | N1—C9—C8 | 109.01 (15) |
| C2—C1—C6 | 119.85 (18) | N1—C9—H9A | 109.9 |
| C2—C1—S1 | 120.41 (15) | C8—C9—H9A | 109.9 |
| C6—C1—S1 | 119.73 (15) | N1—C9—H9B | 109.9 |
| C1—C2—C3 | 119.53 (19) | C8—C9—H9B | 109.9 |
| C1—C2—H2 | 120.2 | H9A—C9—H9B | 108.3 |
| C3—C2—H2 | 120.2 | N1—C10—C11 | 109.43 (15) |
| C4—C3—C2 | 121.46 (18) | N1—C10—H10A | 109.8 |
| C4—C3—H3 | 119.3 | C11—C10—H10A | 109.8 |
| C2—C3—H3 | 119.3 | N1—C10—H10B | 109.8 |
| C3—C4—C5 | 118.49 (18) | C11—C10—H10B | 109.8 |
| C3—C4—C7 | 121.06 (19) | H10A—C10—H10B | 108.2 |
| C5—C4—C7 | 120.4 (2) | O6—C11—O7 | 125.83 (17) |
| C6—C5—C4 | 120.68 (19) | O6—C11—C10 | 123.36 (17) |
| C6—C5—H5 | 119.7 | O7—C11—C10 | 110.79 (16) |

| | | | |
|---|---|---|---|
| O3—S1—C1—C2 | 16.35 (19) | C3—C4—C5—C6 | 0.9 (3) |
| O1—S1—C1—C2 | −105.40 (17) | C7—C4—C5—C6 | −178.42 (18) |
| O2—S1—C1—C2 | 135.94 (16) | C4—C5—C6—C1 | 1.2 (3) |
| O3—S1—C1—C6 | −163.36 (15) | C2—C1—C6—C5 | −2.3 (3) |
| O1—S1—C1—C6 | 74.89 (17) | S1—C1—C6—C5 | 177.39 (15) |
| O2—S1—C1—C6 | −43.77 (18) | C10—N1—C9—C8 | 175.26 (15) |
| C6—C1—C2—C3 | 1.3 (3) | O4—C8—C9—N1 | −5.4 (3) |
| S1—C1—C2—C3 | −178.42 (15) | O5—C8—C9—N1 | 175.76 (15) |
| C1—C2—C3—C4 | 0.9 (3) | C9—N1—C10—C11 | 172.17 (15) |
| C2—C3—C4—C5 | −1.9 (3) | N1—C10—C11—O6 | −6.8 (3) |
| C2—C3—C4—C7 | 177.37 (19) | N1—C10—C11—O7 | 174.56 (14) |

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| N1—H1*N*1···O2$^{i}$ | 0.88 (1) | 2.02 (1) | 2.885 (2) | 167 (2) |
| N1—H1*N*2···O3$^{ii}$ | 0.88 (1) | 2.06 (2) | 2.792 (2) | 140 (2) |
| O5—H5*O*···O1 | 0.84 (1) | 1.79 (1) | 2.607 (2) | 164 (3) |
| O7—H7*O*···O2$^{iii}$ | 0.84 (1) | 1.85 (1) | 2.659 (2) | 160 (3) |

Symmetry codes: (i) −*x*+1, *y*−1/2, −*z*+1/2; (ii) −*x*+1/2, *y*−1/2, *z*; (iii) *x*−1/2, *y*−1, −*z*+1/2.