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Morpholinium 4-amino-5-methoxy-2-methylbenzensulfonate

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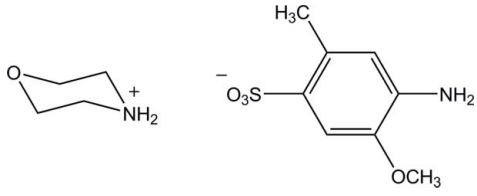
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.002 Å; R factor = 0.044; wR factor = 0.117; data-to-parameter ratio = 16.3.

In the crystal structure of the title compound, $C_4H_{10}NO^+\cdot C_8H_{10}NO_4S^-$, the components are linked by N—H···O hydrogen bonds, forming a centrosymmetric 2:2 aggregate. The aggregates are further connected by N—H···O hydrogen bonds between the anions, forming a double-tape structure along the *a* axis.

Related literature

For related structures, see: Barbour *et al.* (1996); Brito *et al.* (2004); Yin *et al.* (2006).



Experimental

Crystal data

$C_4H_{10}NO^+\cdot C_8H_{10}NO_4S^-$
M_r = 304.37
Monoclinic, $P2_1/c$
a = 9.2141 (5) Å
b = 14.8227 (9) Å
c = 10.4740 (7) Å
β = 91.120 (2)°
V = 1430.24 (15) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.25 mm^{-1}
T = 293 K
0.34 × 0.25 × 0.24 mm

Data collection

Bruker SMART APEX CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2001)
T_{min} = 0.910, T_{max} = 0.931
13039 measured reflections
3105 independent reflections
2962 reflections with $I > 2\sigma(I)$
R_{int} = 0.034

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.044
$wR(F^2)$ = 0.117
S = 1.12
3105 reflections
191 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.27 e Å^{-3}
$\Delta\rho_{min}$ = −0.52 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1A···O2^{i}	0.90	1.92	2.795 (3)	162
N1—H1B···O2	0.90	2.56	3.126 (2)	121
N1—H1B···O3	0.90	1.89	2.790 (3)	175
N2—H2A···O1^{ii}	0.86 (2)	2.23 (2)	3.054 (3)	159.1 (2)
N2—H2B···O5^{iii}	0.85 (2)	2.25 (2)	3.077 (4)	161.5 (2)

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

Data collection: *SMART* (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: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXL97*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: IS2665).

References

Barbour, L. J., Damon, A. K., Orr, G. W. & Atwood, J. L. (1996). *Supramol. Chem.* **7**, 209–211.
Brito, I., Vargas, D., Cardenas, A., Lopez-Rodriguez, M. & Wittke, O. (2004). *J. Chilean Chem. Soc.* **49**, 1–3.
Bruker (2001). *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
Bruker (2007). *SMART* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Yin, C.-X., Huo, F.-J. & Yang, P. (2006). *Acta Cryst.* E**62**, o2084–o2085.

supporting information

Acta Cryst. (2011). E**67**, o631 [doi:10.1107/S1600536811003722]

Morpholinium 4-amino-5-methoxy-2-methylbenzensulfonate

Ling-Gao Shou and Mei-Chao Li

S1. Comment

Several supramolecular structures of morpholinium sulfonate have been reported previously (Barbour *et al.*, 1996; Yin *et al.*, 2006; Brito *et al.*, 2004). As an extension of research, we report here the structure of the title compound, (I).

As shown in Figs. 1 and 2, the 4-amino-5-methoxy-2-methylbenzensulfonate anion is linked to the morpholinium cation by N1—H1B···O3, N1—H1B···O2 and N1—H1A···O2^{i} hydrogen bonds (Table 1). Bond lengths of morpholine ring are very similar to those observed previously (Barbour *et al.*, 1996; Yin *et al.*, 2006; Brito *et al.*, 2004). N2—H2A···O1^{ii} and N2—H2B···O5^{iii} hydrogen bonds (Table 1) also play important roles in stabilizing the crystal.

S2. Experimental

4-Amino-5-methoxy-2-methylbenzensulfonic acid (2.2 g) and morpholine (0.9 g), in a molar ratio of 1:1, were mixed and dissolved in sufficient ethanol by heating to 373 K, at which point a clear solution resulted. The system was then cooled slowly to room temperature. Crystals (2.5 g) were formed, collected and washed with ethanol.

S3. Refinement

H atoms attached to atom N2 were located in a difference Fourier map, and were refined freely. H atoms attached to atom N1 were treated as riding (N—H = 0.90 Å), with U_{iso}(H) = 1.2U_{eq}(N). Other H atoms were placed in calculated positions (C—H = 0.93–0.97 Å), with U_{iso}(H) = 1.2 or 1.5U_{eq}(C).

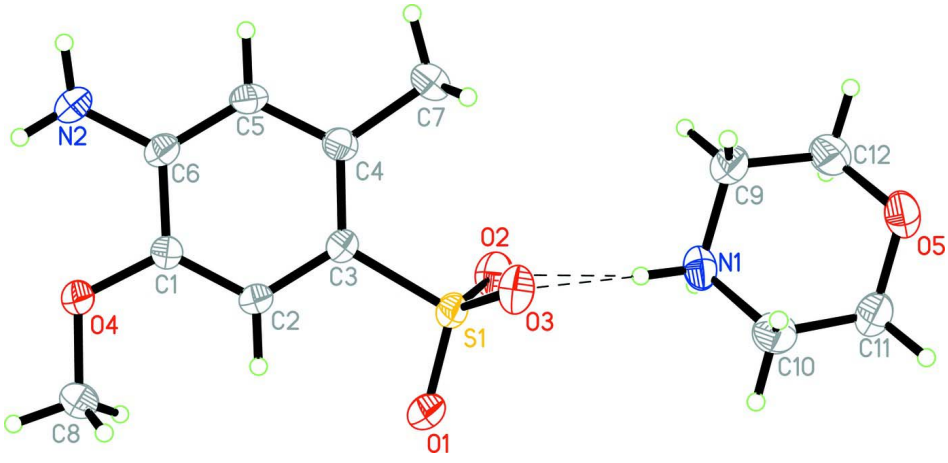


Figure 1

The molecular structure of the title compound, showing the atom-numbering scheme and 50% probability displacement ellipsoids for non-H atoms. Hydrogen bond is illustrated as dashed lines.

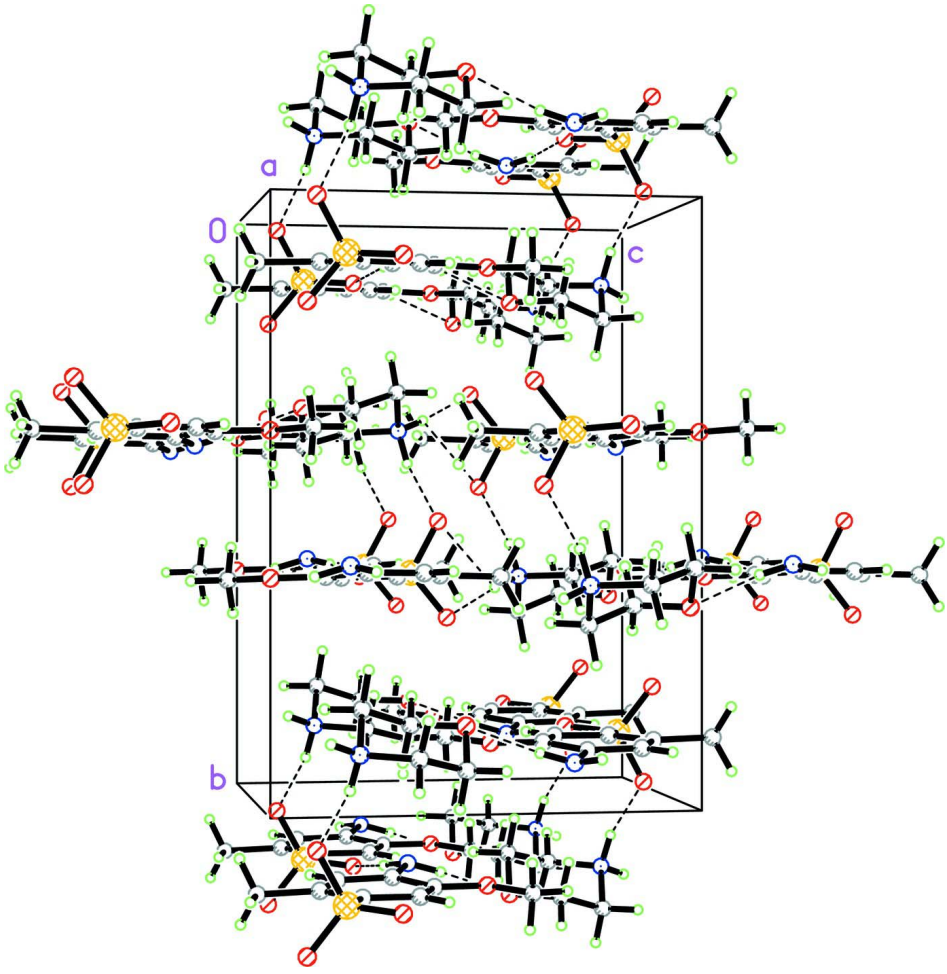


Figure 2

The crystal packing of the title compound, viewed down the *a* axis. Hydrogen bonds are drawn as dashed lines.

Morpholinium 4-amino-5-methoxy-2-methylbenzensulfonate

Crystal data

$C_4H_{10}NO^+·C_8H_{10}NO_4S^-$
M_r = 304.37
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
a = 9.2141 (5) Å
b = 14.8227 (9) Å
c = 10.4740 (7) Å
β = 91.120 (2)°
V = 1430.24 (15) Å^3
Z = 4

$F(000)$ = 648.0
D_x = 1.413 Mg m^{-3}
Melting point: 467 K
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 3615 reflections
θ = 2.3–25.3°
μ = 0.25 mm^{-1}
T = 293 K
Block, colorless
0.34 × 0.25 × 0.24 mm

Data collection

Bruker SMART APEX CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator

φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2001)
T_{min} = 0.910, T_{max} = 0.931

13039 measured reflections
3105 independent reflections
2962 reflections with $I > 2\sigma(I)$
$R_{int} = 0.034$
$\theta_{max} = 27.0°$, $\theta_{min} = 3.2°$
$h = -11 \rightarrow 11$
$k = -18 \rightarrow 18$
$l = -13 \rightarrow 13$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.044$
$wR(F^2) = 0.117$
$S = 1.12$
3105 reflections
191 parameters
0 restraints
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
$w = 1/[\sigma^2(F_o^2) + (0.0661P)^2 + 0.3546P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.27$ e Å^{-3}
$\Delta\rho_{min} = -0.52$ e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 2008), $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.083 (5)

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit *S* are based on F^2, conventional *R*-factors *R* are based on *F*, with *F* set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on *F*, and *R*- factors based on ALL data will be even larger.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
H2A	0.152 (2)	0.1046 (13)	0.8007 (19)	0.044 (5)*
H2B	0.219 (2)	0.1123 (13)	0.925 (2)	0.052 (6)*
C1	0.48589 (15)	0.11584 (9)	0.87597 (13)	0.0312 (3)
C2	0.62334 (14)	0.11986 (9)	0.82610 (13)	0.0316 (3)
H2	0.7039	0.1236	0.8808	0.038*
C3	0.64240 (14)	0.11834 (8)	0.69411 (13)	0.0290 (3)
C4	0.52301 (15)	0.11504 (9)	0.61017 (13)	0.0316 (3)
C5	0.38583 (15)	0.10920 (10)	0.66357 (14)	0.0343 (3)
H5	0.3052	0.1060	0.6090	0.041*
C6	0.36395 (14)	0.10799 (9)	0.79442 (14)	0.0307 (3)
C7	0.53473 (19)	0.11758 (12)	0.46690 (14)	0.0450 (4)
H7A	0.4465	0.0951	0.4284	0.067*
H7B	0.6147	0.0806	0.4414	0.067*
H7C	0.5505	0.1786	0.4397	0.067*
C8	0.57504 (19)	0.12216 (13)	1.09045 (15)	0.0467 (4)
H8C	0.5405	0.1230	1.1763	0.070*
H8D	0.6294	0.1761	1.0748	0.070*
H8E	0.6363	0.0705	1.0790	0.070*
C9	0.91130 (17)	0.12103 (11)	0.23520 (16)	0.0427 (4)

H9A	0.8646	0.1790	0.2222	0.051*
H9B	0.8363	0.0758	0.2453	0.051*
C10	1.12875 (18)	0.18648 (12)	0.33325 (16)	0.0462 (4)
H10A	1.1928	0.1849	0.4078	0.055*
H10B	1.0931	0.2477	0.3228	0.055*
C11	1.21090 (17)	0.15881 (13)	0.21691 (16)	0.0471 (4)
H11A	1.2914	0.1999	0.2047	0.056*
H11B	1.2502	0.0987	0.2293	0.056*
C12	1.0018 (2)	0.09834 (14)	0.12131 (16)	0.0526 (4)
H12A	1.0400	0.0377	0.1310	0.063*
H12B	0.9408	0.0996	0.0448	0.063*
O1	0.91647 (12)	0.12197 (9)	0.75110 (12)	0.0501 (3)
O2	0.83977 (12)	0.02929 (7)	0.57210 (11)	0.0449 (3)
O3	0.83980 (11)	0.19031 (7)	0.55105 (10)	0.0416 (3)
O5	1.11893 (13)	0.15976 (10)	0.10715 (11)	0.0533 (3)
O4	0.45499 (12)	0.11751 (9)	1.00344 (10)	0.0447 (3)
N1	1.00509 (15)	0.12414 (9)	0.35140 (12)	0.0383 (3)
H1A	1.0389	0.0685	0.3690	0.046*
H1B	0.9527	0.1427	0.4181	0.046*
N2	0.22873 (14)	0.09542 (10)	0.84692 (15)	0.0416 (3)
S1	0.82261 (3)	0.11518 (2)	0.63924 (3)	0.03170 (15)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0266 (7)	0.0409 (7)	0.0263 (6)	0.0017 (5)	0.0007 (5)	−0.0003 (5)
C2	0.0234 (6)	0.0425 (7)	0.0287 (6)	0.0004 (5)	−0.0023 (5)	0.0000 (5)
C3	0.0247 (6)	0.0331 (6)	0.0292 (6)	0.0013 (4)	0.0016 (5)	−0.0007 (5)
C4	0.0310 (7)	0.0359 (7)	0.0278 (6)	0.0026 (5)	−0.0022 (5)	−0.0020 (5)
C5	0.0261 (6)	0.0428 (8)	0.0337 (7)	0.0015 (5)	−0.0068 (5)	−0.0025 (5)
C6	0.0240 (6)	0.0330 (6)	0.0351 (7)	0.0022 (5)	−0.0005 (5)	−0.0005 (5)
C7	0.0442 (9)	0.0628 (10)	0.0279 (7)	0.0011 (7)	−0.0031 (6)	−0.0029 (6)
C8	0.0391 (8)	0.0722 (11)	0.0285 (7)	0.0002 (7)	−0.0041 (6)	0.0019 (7)
C9	0.0326 (8)	0.0515 (9)	0.0441 (9)	0.0013 (6)	0.0020 (6)	0.0035 (6)
C10	0.0465 (9)	0.0476 (9)	0.0444 (8)	−0.0033 (7)	−0.0013 (7)	−0.0057 (7)
C11	0.0357 (7)	0.0563 (10)	0.0495 (9)	−0.0041 (7)	0.0074 (7)	0.0044 (7)
C12	0.0480 (10)	0.0733 (12)	0.0366 (8)	−0.0010 (8)	0.0003 (7)	−0.0092 (8)
O1	0.0255 (5)	0.0828 (9)	0.0420 (6)	−0.0007 (5)	−0.0006 (5)	0.0016 (5)
O2	0.0445 (6)	0.0391 (6)	0.0515 (6)	0.0111 (4)	0.0101 (5)	−0.0029 (5)
O3	0.0421 (6)	0.0389 (6)	0.0442 (6)	−0.0001 (4)	0.0136 (5)	0.0033 (4)
O5	0.0477 (7)	0.0754 (9)	0.0371 (6)	0.0013 (6)	0.0099 (5)	0.0109 (6)
O4	0.0280 (5)	0.0799 (9)	0.0263 (5)	0.0004 (5)	0.0018 (4)	0.0003 (5)
N1	0.0404 (7)	0.0415 (7)	0.0336 (6)	0.0080 (5)	0.0096 (5)	0.0055 (5)
N2	0.0233 (6)	0.0583 (8)	0.0431 (7)	0.0010 (5)	0.0007 (5)	−0.0006 (6)
S1	0.0252 (2)	0.0379 (2)	0.0322 (2)	0.00271 (11)	0.00416 (14)	0.00068 (12)

Geometric parameters (Å, º)

C1—O4	1.3707 (17)	C9—C12	1.507 (2)
C1—C2	1.3806 (19)	C9—H9A	0.9700
C1—C6	1.4029 (19)	C9—H9B	0.9700
C2—C3	1.3972 (19)	C10—N1	1.482 (2)
C2—H2	0.9300	C10—C11	1.504 (2)
C3—C4	1.3955 (19)	C10—H10A	0.9700
C3—S1	1.7683 (13)	C10—H10B	0.9700
C4—C5	1.395 (2)	C11—O5	1.415 (2)
C4—C7	1.507 (2)	C11—H11A	0.9700
C5—C6	1.389 (2)	C11—H11B	0.9700
C5—H5	0.9300	C12—O5	1.422 (2)
C6—N2	1.3842 (18)	C12—H12A	0.9700
C7—H7A	0.9600	C12—H12B	0.9700
C7—H7B	0.9600	O1—S1	1.4461 (12)
C7—H7C	0.9600	O2—S1	1.4645 (11)
C8—O4	1.4211 (18)	O3—S1	1.4575 (11)
C8—H8C	0.9600	N1—H1A	0.9000
C8—H8D	0.9600	N1—H1B	0.9000
C8—H8E	0.9600	N2—H2A	0.86 (2)
C9—N1	1.480 (2)	N2—H2B	0.86 (2)
O4—C1—C2	125.26 (12)	N1—C10—C11	109.53 (13)
O4—C1—C6	114.54 (12)	N1—C10—H10A	109.8
C2—C1—C6	120.19 (12)	C11—C10—H10A	109.8
C1—C2—C3	120.51 (12)	N1—C10—H10B	109.8
C1—C2—H2	119.7	C11—C10—H10B	109.8
C3—C2—H2	119.7	H10A—C10—H10B	108.2
C4—C3—C2	120.74 (12)	O5—C11—C10	110.65 (13)
C4—C3—S1	121.87 (11)	O5—C11—H11A	109.5
C2—C3—S1	117.32 (10)	C10—C11—H11A	109.5
C5—C4—C3	117.32 (12)	O5—C11—H11B	109.5
C5—C4—C7	118.94 (13)	C10—C11—H11B	109.5
C3—C4—C7	123.74 (13)	H11A—C11—H11B	108.1
C6—C5—C4	123.13 (12)	O5—C12—C9	111.88 (15)
C6—C5—H5	118.4	O5—C12—H12A	109.2
C4—C5—H5	118.4	C9—C12—H12A	109.2
N2—C6—C5	122.87 (13)	O5—C12—H12B	109.2
N2—C6—C1	119.06 (13)	C9—C12—H12B	109.2
C5—C6—C1	118.01 (13)	H12A—C12—H12B	107.9
C4—C7—H7A	109.5	C11—O5—C12	110.65 (12)
C4—C7—H7B	109.5	C1—O4—C8	116.84 (12)
H7A—C7—H7B	109.5	C9—N1—C10	110.61 (12)
C4—C7—H7C	109.5	C9—N1—H1A	109.5
H7A—C7—H7C	109.5	C10—N1—H1A	109.5
H7B—C7—H7C	109.5	C9—N1—H1B	109.5
O4—C8—H8C	109.5	C10—N1—H1B	109.5

O4—C8—H8D	109.5	H1A—N1—H1B	108.1
H8C—C8—H8D	109.5	C6—N2—H2A	119.5 (13)
O4—C8—H8E	109.5	C6—N2—H2B	116.8 (15)
H8C—C8—H8E	109.5	H2A—N2—H2B	112.7 (19)
H8D—C8—H8E	109.5	O1—S1—O3	112.93 (7)
N1—C9—C12	109.57 (13)	O1—S1—O2	112.40 (7)
N1—C9—H9A	109.8	O3—S1—O2	110.23 (7)
C12—C9—H9A	109.8	O1—S1—C3	106.58 (7)
N1—C9—H9B	109.8	O3—S1—C3	107.47 (6)
C12—C9—H9B	109.8	O2—S1—C3	106.86 (6)
H9A—C9—H9B	108.2		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*A*···O2^{i}	0.90	1.92	2.795 (3)	162
N1—H1*B*···O2	0.90	2.56	3.126 (2)	121
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N2—H2*B*···O5^{iii}	0.85 (2)	2.25 (2)	3.077 (4)	161.5 (2)

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