are 2·05 (4), 1·96 (7) and 2·05 (7) Å respectively and the respective angles N—H···O are 173 (4), 149 (6) and 164 (7)°. Such $COO^-\cdots H_3N^+$ hydrogen bonds are found in all amino acids. There are, however, no short contacts with the Se atom, though selenides have been shown to form short contacts with electrophiles and nucleophiles (Ramasubbu & Parthasarathy, 1983) as in the case of sulfides (Rosenfield, Parthasarathy & Dunitz, 1977).

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Structure of 2,4-Diamino-5-(3,4,5-trimethoxybenzyl)pyrimidinium Benzoate (Trimethoprim Monobenzoate), $C_{14}H_{19}N_4O_3^+.C_7H_5O_2^-$

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Abstract. $M_r = 412\cdot4$, monoclinic, $P2_1/n$, $a = 11\cdot295$ (1), $b = 28\cdot266$ (2), $c = 6\cdot543$ (1) Å, $\beta = 100\cdot97$ (1)°, $V = 2050\cdot8$ (4) Å^3, $Z = 4$, $D_m = 1\cdot36$, $D_x = 1\cdot336$ Mg m^{-3}, Cu $K\alpha$, $\lambda = 1\cdot5418$ Å, $\mu = 0\cdot81$ mm^{-1}, $F(000) = 872$, $T = 296$ K, $R = 0\cdot039$ for 1895 observed reflections. The interaction compound is formed by the trimethoprim N(1) cation and the benzoate anion. Two interionic hydrogen bonds lead to the formation of an eight-membered pseudo ring. Cyclic dimers *via* pairs of hydrogen bonds across centers of symmetry are formed by the ions of the title compound.

Introduction. Binary systems in which the antifolate drug trimethoprim [2,4-diamino-5-(3,4,5-trimethoxybenzyl)pyrimidine, TMP] is involved have been investigated by some of us both for phase equilibria and for thermal behavior. TMP and sulfamethoxazole (SMZ), components of widely used antibacterial formulations (*e.g.* BACTRIM^R), give a 1:1 molecular compound, TSC, with a congruent melting point (Giordano, Bettinetti, La Manna & Ferloni, 1977). This compound, totally dissociated in the melt (Margheritis & Sinistri, 1978), is a hydrogen-bonded complex (Giuseppetti, Tadini, Bettinetti, Giordano & La Manna, 1980). TMP and benzoic acid (BA) form in the solid phase two congruent melting intermediate compounds, BA–TMP and $(BA)_2$–TMP, which show only partial dissociation in the melt (Bettinetti, Caramella, Giordano, La Manna, Margheritis & Sinistri, 1983). The high stability of these interaction compounds suggests a bond pattern stronger than that found by us in the TMP–SMZ complex.

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This crystallographic study was undertaken in order to elucidate the way in which TMP and BA are linked in BA–TMP. Moreover, this investigation could provide data to allow structural relationships between the TMP molecule in the free solid state (Koetzle & Williams, 1976) and in the complexed solid states (Giuseppetti, Tadini, Bettinetti, Giordano & La Manna, 1980; Haltiwanger, 1971) to be made.

Experimental. Single crystals obtained by slow evaporation of a solution in ethyl acetate of equimolar amounts of BA and TMP; density measured with a helium pycnometer model 1302/1303 Micromeritics Instrument Corp.; prismatic crystal (0·35 × 0·25 × 0·22 mm); Philips PW 1100 four-circle diffractometer, graphite monochromator; 25 reflections ($2 < \theta < 45°$) used for measuring lattice parameters with Philips *LAT* routine; θ–2θ scan mode, scan speed 0·05° s^{-1}, scan width 1·0°, θ range 2–55°, three standard reflections every 180 min, mean intensity variation 2·2%; 2564 independent data ($-11 \leq h \leq 11$, $k \leq 30$, $l \leq 6$, max. sin θ/λ 0·53 $Å^{-1}$), 1895 observed with $I > 3\sigma(I)$, corrections for Lorentz–polarization and experimental absorption, between 1·0003 and 1·1391, following North, Phillips & Mathews (1968). Structure solved by direct methods (*MULTAN*78; Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978); F magnitudes used in least-squares refinement; coordinates of H atoms calculated by geometrical considerations (*XANADU*, Roberts & Sheldrick, 1975), confirmed by ΔF synthesis showing random fluctuations between 0·13 and −0·16 e $Å^{-3}$; parameters refined: coordinates and anisotropic thermal parameters for non-hydrogen atoms, hydrogen atoms with isotropic temperature factors, scale and secondary extinction [final $g = 1{\cdot}24\,(2) \times 10^{-3}$ calculated anisotropic following Coppens & Hamilton (1970)] coefficients. Final value of $R_{\rm obs} = 3{\cdot}87\%$, $R_{\rm all} = 5{\cdot}32\%$, unit weights, $S = 0{\cdot}723$; max. Δ/σ in the last cycle of refinement was 0·036; atomic scattering factors for neutral atoms taken from *International Tables for X-ray Crystallography* (1974); a locally modified version of program *ORFLS* (Busing, Martin & Levy, 1962) and the program *PARST* (Nardelli, 1983) were used. Figures were drawn with the *ORTEP*II program (Johnson, 1976).

Discussion. Final atomic coordinates and equivalent isotropic temperature factors are given in Table 1.* Bond distances and angles are listed in Table 2. As shown in Fig. 1(*a*), the crystalline compound BA–TMP has a well defined salt character involving a protonated cation from TMP [N(1)] and the benzoate anion. Ions are linked by a strong hydrogen-bond-type interaction $^{(+)}$N(1)–H(11)···$^{(-)}$O(11,1) and a weaker interaction N(2)–H2(N2)···O(11,2). The $^{(+)}$N(1)···$^{(-)}$O(11,1) separation of 2·578 (3) Å is significantly shorter than the N(2)···O(11,2) separation of 2·813 (3) Å owing to a major electrostatic interaction. H(11)···$^{(-)}$O(11,1) and H2(N2)···O(11,2) distances are 1·57 (3) and 1·89 (3) Å, respectively.

Table 1. *Fractional atomic coordinates* ($\times 10^4$ *for non-hydrogen atoms,* $\times 10^3$ *for hydrogen atoms) and equivalent isotropic temperature factors* ($Å^2$), *with e.s.d.'s in parentheses*

$$B_{\rm eq} = \tfrac{1}{3}\textstyle\sum_i \sum_j B_{ij} a_i^* a_j^* \mathbf{a}_i . \mathbf{a}_j .$$

	x	y	z	$B_{\rm eq}/B_{\rm iso}$
N(1)	6099 (2)	4556 (1)	10298 (3)	3·62 (5)
C(2)	5230 (2)	4386 (1)	11272 (4)	3·48 (7)
N(3)	4585 (2)	4003 (1)	10629 (3)	3·81 (5)
C(4)	4791 (2)	3786 (1)	8919 (4)	3·56 (7)
C(5)	5692 (2)	3939 (1)	7806 (4)	3·21 (7)
C(6)	6312 (2)	4326 (1)	8567 (4)	3·55 (7)
C(7)	5936 (3)	3681 (1)	5909 (4)	3·58 (8)
N(2)	5047 (3)	4618 (1)	12954 (4)	4·21 (7)
N(4)	4117 (2)	3408 (1)	8272 (4)	5·15 (7)
C(1*a*)	6493 (2)	3193 (1)	6341 (4)	3·20 (7)
C(2*a*)	6230 (2)	2850 (1)	4811 (4)	3·46 (7)
C(3*a*)	6746 (2)	2407 (1)	5132 (4)	3·47 (7)
C(4*a*)	7521 (2)	2299 (1)	6999 (4)	3·32 (6)
C(5*a*)	7766 (2)	2642 (1)	8529 (4)	3·27 (5)
C(6*a*)	7261 (2)	3089 (1)	8213 (4)	3·28 (7)
O(3*a*)	6564 (2)	2043 (1)	3727 (3)	4·90 (5)
O(4*a*)	8088 (2)	1866 (1)	7273 (3)	4·24 (5)
O(5*a*)	8521 (2)	2501 (1)	10325 (3)	4·54 (4)
C(8)	8947 (4)	2854 (1)	11841 (5)	5·46 (14)
C(9)	7360 (4)	1478 (1)	7722 (7)	5·15 (10)
C(10)	5751 (3)	2126 (1)	1794 (5)	4·96 (13)
O(11,1)	7468 (2)	5260 (1)	11682 (3)	5·84 (5)
O(11,2)	6874 (2)	5252 (1)	14737 (4)	7·65 (6)
C(11)	7629 (3)	5342 (1)	13617 (6)	5·13 (9)
C(12)	8830 (3)	5542 (1)	14602 (5)	4·49 (8)
C(13)	9556 (3)	5759 (1)	13395 (6)	5·62 (10)
C(14	10676 (3)	5935 (1)	14289 (7)	6·45 (16)
C(15)	11097 (3)	5888 (1)	16399 (7)	6·55 (16)
C(16)	10386 (3)	5671 (1)	17608 (7)	6·70 (15)
C(17)	9250 (3)	5501 (1)	16734 (6)	5·60 (10)
H(11)	656 (3)	486 (1)	1079 (4)	3·5 (7)
H(6)	697 (2)	447 (1)	794 (3)	0·8 (5)
H1(N2)	436 (3)	456 (1)	1348 (5)	2·5 (7)
H2(N2)	553 (3)	489 (1)	1342 (5)	3·9 (8)
H1(N4)	367 (3)	333 (1)	883 (5)	1·4 (7)
H2(N4)	414 (3)	322 (1)	702 (5)	4·2 (8)
H(7,1)	518 (3)	364 (1)	477 (4)	2·5 (7)
H(7,2)	650 (2)	389 (1)	527 (4)	1·5 (6)
H(2*a*)	569 (2)	293 (1)	346 (4)	1·6 (6)
H(6*a*)	748 (2)	333 (1)	923 (4)	1·3 (6)
H(8,1)	835 (3)	299 (1)	1243 (5)	3·1 (8)
H(8,2)	928 (3)	315 (1)	1123 (5)	4·3 (9)
H(8,3)	953 (3)	268 (1)	1296 (5)	4·6 (9)
H(9,1)	776 (3)	118 (1)	767 (6)	6·2 (10)
H(9,2)	660 (4)	146 (1)	661 (7)	7·8 (13)
H(9,3)	725 (4)	152 (1)	901 (8)	9·4 (17)
H(10,1)	568 (3)	178 (1)	110 (5)	4·0 (8)
H(10,2)	497 (3)	220 (1)	204 (4)	2·2 (7)
H(10,3)	599 (3)	241 (1)	106 (5)	3·4 (8)
H(13)	923 (4)	580 (1)	1188 (5)	3·6 (8)
H(14)	1122 (3)	612 (1)	1343 (6)	7·0 (10)
H(15)	1197 (4)	601 (1)	1710 (6)	7·1 (11)
H(16)	1070 (3)	561 (1)	1913 (6)	6·1 (10)
H(17)	868 (3)	533 (1)	1754 (5)	3·9 (8)

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* Lists of structure factors, anisotropic temperature factors for non-hydrogen atoms, torsion angles, least-squares planes with the deviations of the atoms from the planes and some intermolecular contacts ranging from 3·00 to 3·22 Å, and a figure showing molecular packing and other intermolecular contacts have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 39052 (24 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

The eight-atom pseudo-ring system generated by the linkage between TMP and BA is represented in detail in Fig. 1(*b*) and the analysis of ring-puckering coordinates following Cremer & Pople (1975) leads to a pseudo twist-boat conformation [total puckering amplitude $Q = 0{\cdot}435\ (19)$ Å], which is confirmed by the displacements of atoms relative to the mean plane.

The BA–TMP ion couples are linked to produce cyclic dimers *via* pairs of N(2)–H1(N2)···O(11,2) hydrogen bonds across centers of symmetry (Fig. 2).*

The stability of the crystalline complex BA–TMP, as evidenced by its low dissociation in the melt (Bettinetti, Caramella, Giordano, La Manna, Margheritis & Sinistri, 1983), is hence attributable to the ionic character of the bond between BA and TMP and to the packing forces between its molecules.

When the TMP cation in BA–TMP is considered, the main structural effect of protonation with respect to TMP is to increase substantially the ring angle at the N(1) atom, ∠C(2)–N(1)–C(6), from 115·46 (5)° (Koetzle & Williams, 1976) to 118·7 (2)°. The endocyclic angles adjacent to the site of addition are

Table 2. *Bond distances* (Å) *and angles* (°) *with e.s.d.'s in parentheses*

N(1)–C(2)	1·355 (4)	O(3*a*)–C(10)	1·433 (4)
N(1)–C(6)	1·366 (4)	O(4*a*)–C(9)	1·435 (5)
N(1)–H(11)	1·02 (3)	O(5*a*)–C(8)	1·424 (4)
C(2)–N(3)	1·329 (4)	C(8)–H(8,1)	0·92 (3)
C(2)–N(2)	1·331 (4)	C(8)–H(8,2)	1·04 (3)
N(3)–C(4)	1·334 (4)	C(8)–H(8,3)	1·01 (3)
C(4)–C(5)	1·426 (4)	C(9)–H(9,1)	0·96 (3)
C(4)–N(4)	1·333 (4)	C(9)–H(9,2)	1·02 (4)
C(5)–C(6)	1·344 (4)	C(9)–H(9,3)	0·88 (5)
C(5)–C(7)	1·509 (4)	C(10)–H(10,1)	1·07 (3)
C(6)–H(6)	1·00 (2)	C(10)–H(10,2)	0·95 (3)
N(2)–H1(N2)	0·92 (4)	C(10)–H(10,3)	1·00 (3)
N(2)–H2(N2)	0·96 (3)	O(11,1)–C(11)	1·265 (4)
N(4)–H1(N4)	0·71 (4)	O(11,1)–H(11)	1·57 (3)
N(4)–H2(N4)	0·99 (3)	O(11,2)–H2(N2)	1·89 (2)
C(7)–C(1*a*)	1·520 (4)	O(11,2)–C(11)	1·251 (5)
C(7)–H(7,1)	1·03 (3)	C(11)–C(12)	1·498 (4)
C(7)–H(7,2)	1·01 (3)	C(12)–C(13)	1·384 (5)
C(1*a*)–C(2*a*)	1·386 (4)	C(12)–C(17)	1·390 (5)
C(1*a*)–C(6*a*)	1·392 (3)	C(13)–C(14)	1·382 (4)
C(2*a*)–C(3*a*)	1·379 (4)	C(13)–H(13)	1·00 (3)
C(2*a*)–H(2*a*)	1·00 (2)	C(14)–C(15)	1·379 (6)
C(3*a*)–C(4*a*)	1·394 (3)	C(14)–H(14)	1·05 (4)
C(3*a*)–O(3*a*)	1·369 (4)	C(15)–C(16)	1·374 (6)
C(4*a*)–C(5*a*)	1·383 (4)	C(15)–H(15)	1·06 (4)
C(4*a*)–O(4*a*)	1·378 (4)	C(16)–C(17)	1·388 (4)
C(5*a*)–C(6*a*)	1·386 (4)	C(16)–H(16)	1·00 (4)
C(5*a*)–O(5*a*)	1·373 (3)	C(17)–H(17)	1·03 (3)
C(6*a*)–H(6*a*)	0·95 (3)		
C(2)–N(1)–C(6)	118·7 (2)	C(3*a*)–O(3*a*)–C(10)	117·3 (2)
C(2)–N(1)–H(11)	122 (2)	C(4*a*)–O(4*a*)–C(9)	115·8 (2)
C(6)–N(1)–H(11)	120 (2)	C(5*a*)–O(5*a*)–C(8)	117·7 (2)
N(1)–C(2)–N(3)	122·8 (2)	O(5*a*)–C(8)–H(8,1)	114 (2)
N(1)–C(2)–N(2)	117·0 (2)	O(5*a*)–C(8)–H(8,2)	114 (2)
N(3)–C(2)–N(2)	120·1 (2)	H(8,1)–C(8)–H(8,3)	109 (3)
C(2)–N(3)–C(4)	117·8 (2)	O(5*a*)–C(8)–H(8,3)	104 (2)
N(3)–C(4)–C(5)	123·0 (2)	H(8,2)–C(8)–H(8,3)	116 (3)
N(3)–C(4)–N(4)	117·0 (2)	H(8,1)–C(8)–H(8,2)	99 (2)
C(5)–C(4)–N(4)	120·0 (2)	O(4*a*)–C(9)–H(9,2)	109 (2)
C(4)–C(5)–C(6)	115·5 (2)	O(4*a*)–C(9)–H(9,3)	107 (2)
C(4)–C(5)–C(7)	122·2 (2)	O(4*a*)–C(9)–H(9,1)	112 (2)
C(6)–C(5)–C(7)	122·3 (2)	H(9,1)–C(9)–H(9,2)	106 (3)
N(1)–C(6)–C(5)	122·1 (2)	H(9,1)–C(9)–H(9,3)	107 (3)
N(1)–C(6)–H(6)	114 (1)	H(9,2)–C(9)–H(9,3)	116 (4)
C(5)–C(6)–H(6)	123 (1)	O(3*a*)–C(10)–H(10,1)	102 (2)
C(2)–N(2)–H1(N2)	119 (2)	O(3*a*)–C(10)–H(10,2)	110 (2)
C(2)–N(2)–H2(N2)	119 (2)	O(3*a*)–C(10)–H(10,3)	112 (2)
H1(N2)–N(2)–H2(N2)	120 (3)	H(10,1)–C(10)–H(10,3)	122 (2)
C(4)–N(4)–H1(N4)	120 (2)	H(10,2)–C(10)–H(10,3)	105 (2)
C(4)–N(4)–H2(N4)	127 (2)	H(10,1)–C(10)–H(10,2)	106 (2)
H1(N4)–N(4)–H2(N4)	113 (3)	C(11)–O(11,1)–H(11)	118 (1)
C(5)–C(7)–H(7,2)	107 (2)	C(11)–O(11,2)–H2(N2)	115 (1)
C(5)–C(7)–C(1*a*)	114·8 (2)	O(11,1)–C(11)–O(11,2)	124·4 (3)
C(5)–C(7)–H(7,1)	113 (2)	C(12)–C(11)–O(11,1)	116·4 (3)
C(1*a*)–C(7)–H(7,2)	109 (1)	O(11,2)–C(11)–C(12)	119·2 (3)
H(7,1)–C(7)–H(7,2)	105 (2)	C(11)–C(12)–C(13)	120·5 (3)
C(1*a*)–C(7)–H(7,1)	107 (2)	C(11)–C(12)–C(17)	120·5 (3)
C(2*a*)–C(1*a*)–C(6*a*)	120·0 (2)	C(13)–C(12)–C(17)	118·9 (3)
C(7)–C(1*a*)–C(2*a*)	118·5 (2)	C(12)–C(13)–C(14)	120·8 (3)
C(7)–C(1*a*)–C(6*a*)	121·5 (2)	C(12)–C(13)–H(13)	118 (2)
C(1*a*)–C(2*a*)–C(3*a*)	120·0 (2)	C(14)–C(13)–H(13)	121 (2)
C(1*a*)–C(2*a*)–H(2*a*)	119 (1)	C(13)–C(14)–C(15)	120·2 (3)
C(3*a*)–C(2*a*)–H(2*a*)	121 (1)	C(13)–C(14)–H(14)	122 (2)
C(2*a*)–C(3*a*)–C(4*a*)	120·6 (2)	C(15)–C(14)–H(14)	117 (2)
C(4*a*)–C(3*a*)–O(3*a*)	114·4 (2)	C(14)–C(15)–C(16)	119·5 (3)
C(2*a*)–C(3*a*)–O(3*a*)	125·0 (2)	C(14)–C(15)–H(15)	121 (2)
C(3*a*)–C(4*a*)–C(5*a*)	119·2 (2)	C(16)–C(15)–H(15)	119 (2)
C(3*a*)–C(4*a*)–O(4*a*)	120·5 (2)	C(15)–C(16)–C(17)	120·9 (3)
C(5*a*)–C(4*a*)–O(4*a*)	120·2 (2)	C(15)–C(16)–H(16)	121 (2)
C(4*a*)–C(5*a*)–C(6*a*)	120·7 (2)	C(17)–C(16)–H(16)	118 (2)
C(4*a*)–C(5*a*)–O(5*a*)	115·0 (2)	C(12)–C(17)–C(16)	119·8 (3)
C(6*a*)–C(5*a*)–O(5*a*)	124·3 (2)	C(16)–C(17)–H(17)	125 (1)
C(1*a*)–C(6*a*)–C(5*a*)	120·0 (2)	C(12)–C(17)–H(17)	115 (1)
C(1*a*)–C(6*a*)–H(6*a*)	120 (1)	O(11,1)–H(11)–N(1)	170 (3)
C(5*a*)–C(6*a*)–H(6*a*)	120 (1)	O(11,2)–H2(N2)–N(2)	158 (2)

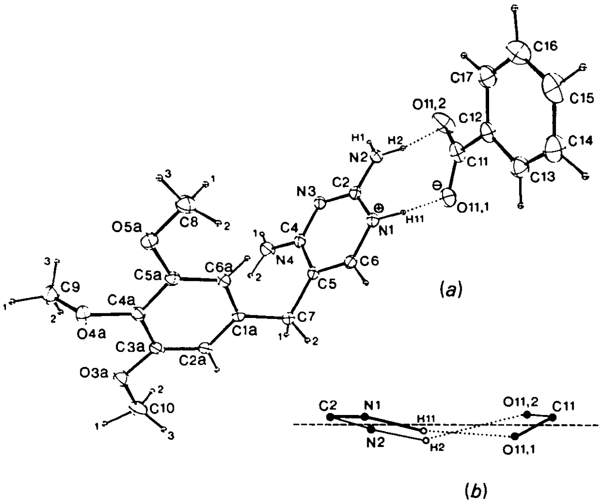

Fig. 1. (*a*) Molecular conformation of BA–TMP: the H to O interactions between the TMP cation and the carboxylate anion are shown by broken lines. (*b*) Eight-atom pseudo-ring system generated by the linkage between TMP and BA.

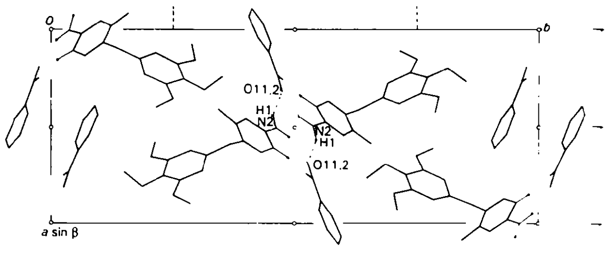

Fig. 2. View along **c** showing packing of molecules *via* pairs of N(2)–H1(N2)···O(11,2) hydrogen bonds. N(2)···O(11,2): 2·894 (3) Å; H(1)···O(11,2): 2·06 (3) Å; N(2)–H(1)···O(11,2): 150 (4)°. The symmetry-related positions are: N(2) and H1(N2): *x*, *y*, *z*; O(11,2): 1 − *x*, 1 − *y*, 3 − *z*.

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* See deposition footnote.

correspondingly adjusted, *i.e.* diminished from about 125° in TMP to 122–123° in its cation. Another effect of the protonation is to increase the length of the ring bonds adjacent to the N(1) atom from 1·343 (1) Å (the value of both bond lengths in TMP) to 1·355 (4) Å for N(1)–C(2) and to 1·366 (4) Å for N(1)–C(6). The conformation adopted by the TMP cation is described by the torsion angles $\tau_1 = \mathrm{C(4)–C(5)–C(7)–C(1}a)$ of −68·4 (3)° and $\tau_2 = \mathrm{C(5)–C(7)–C(1}a\mathrm{)–C(2}a)$ of 149·1 (2)°. TMP (Koetzle & Williams, 1976) and TMP acetate (Haltiwanger, 1971) adopt similar conformations in the solid state [$\tau_{1(\mathrm{TMP})} = -89{\cdot}4$ (1); $\tau_{2(\mathrm{TMP})} = 153{\cdot}3$ (1); $\tau_{1(\mathrm{TMP.Ac})} = -77{\cdot}9$, $\tau_{2(\mathrm{TMP.Ac})} = 155{\cdot}1$°], while the conformation of the complex TMP–SMZ is different [$\tau_{1(\mathrm{TSC})} = 173{\cdot}4$ (4); $\tau_{2(\mathrm{TSC})} = 87{\cdot}7$ (4)°].

The structural and steric situation of the TMP benzene ring and of the benzoate anion is the same as observed in analogous compounds.

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1,2,5-Telluradiazole, $C_2H_2N_2Te$

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Abstract. $M_r = 181{\cdot}65$, orthorhombic, *Pnma*, $a = 12{\cdot}308$ (2), $b = 7{\cdot}840$ (2), $c = 4{\cdot}136$ (1) Å, $V = 399{\cdot}1$ (4) Å^3, $Z = 4$, D_m (by flotation) = 3·01, $D_x = 3{\cdot}022$ g cm^{-3}, Mo $K\alpha$, $\lambda = 0{\cdot}71069$ Å, $\mu = 72{\cdot}43$ cm^{-1}, $F(000) = 320$, $T = 293$ K, final conventional R is 0·031 for 339 unique observed reflections. The title compound has been obtained by transformation of the 1,2,5-selenadiazole into the tellurium isologue. The molecule is a heterocycle which contains the bonds N–Te–N, but the tellurium atom forms two other weak bonds with two nitrogen atoms of different molecules. In this way a ribbon polymeric structure is produced which accounts for the exceptionally high melting point and low solubility in common solvents of the compound.

Introduction. When the conversion of 1,2,5-selenadiazoles or 1,2,5-thiadiazoles into the corresponding 1,2,5-telluradiazoles allowed for the first time the synthesis of such derivatives (Bertini, Lucchesini & De Munno, 1982), we were surprised by their unusual physico-chemical properties. The 1,2,5-telluradiazole as

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