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Jesromotetrol

By Volker Zabel and William H. Watson*

FASTBIOS Laboratory, Department of Chemistry, Texas Christian University, Fort Worth, Texas 76129, *USA*

and Xorge A. Domínguez, Carmen Cisneros, Elsa Guajardo and Romelia Villarreal

Departamento de Química, ITESM, Monterrey, NL, Mexico

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Abstract. $C_{20}H_{34}O_4.H_2O$, $M_r = 356{\cdot}51$, monoclinic, $P2_1$, $a = 26{\cdot}48$ (1), $b = 6{\cdot}622$ (3), $c = 11{\cdot}550$ (4) Å, $\beta = 104{\cdot}67$ (3)°, $V = 1959$ (1) Å^3, $\mu = 0{\cdot}692$ mm^{-1}, $Z = 4$, $d_c = 1{\cdot}209$ Mg m^{-3}. Full-matrix least-squares refinement (nonhydrogen atoms anisotropic, H atoms isotropic) based on 3373 reflexions led to a final R of 0·048. The three six-membered rings of the rosane-type diterpene exist in flattened-chair, half-chair and chair conformations with a *trans* B/C ring juncture. The molecules are held together by a hydrogen-bonding network involving water molecules.

Introduction. It is difficult to decide whether members of the *Palafoxia* genus (Compositae) should be placed within the tribe Heliantheae or the tribe Eupatorieae. For phyletic reasons they are considered to be members of the Eupatorieae and sole members of the subtribe Palafoxiinae (Turner & Morris, 1976). It is anticipated that phytochemical data will provide independent evidence for the placement of this genus. *Palafoxia rosea* is a native of Mexico and has been used in the Tampico area as a folk medicine for the treatment of fever, nausea and chills (Martinez, 1969). *P. rosea* has yielded several new labdane-type terpenes (Dominguez, Cisneros, Guajardo, Villarreal & Zamudio, 1975) and jesromotetrol, a diterpene with a rosane-type skeleton (13-*epi*-rimuene) (Dominguez, Cisneros, Guajardo, Villarreal, Zabel & Watson, 1978). The structure of jesromotetrol was elucidated by X-ray diffraction techniques.

A crystal of dimensions 0·07 × 0·50 × 0·50 mm was used to collect all data on a Syntex $P2_1$ diffractometer system by the θ:2θ scanning technique with a variable scan speed and Cu $K\alpha$ radiation ($\lambda = 1{\cdot}54178$ Å) and a graphite monochromator. Room-temperature lattice parameters were refined by a least-squares procedure utilizing 15 reflexions whose angles were measured by a centering routine associated with the Syntex diffractometer. Systematic absences and optical activity were consistent with space group $P2_1$. Of the 3565 independent reflexions measured ($2\theta < 140°$), 3373 had intensities greater than $2\sigma(I)$. A standard reflexion was monitored and no

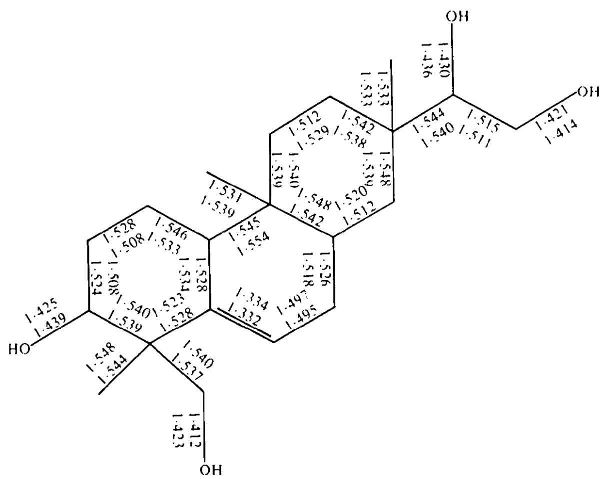

Fig. 1. Bond lengths (Å) for the two independent jesromotetrol molecules (standard deviations 0·003 or 0·004 Å). The upper numbers correspond to molecule 1.

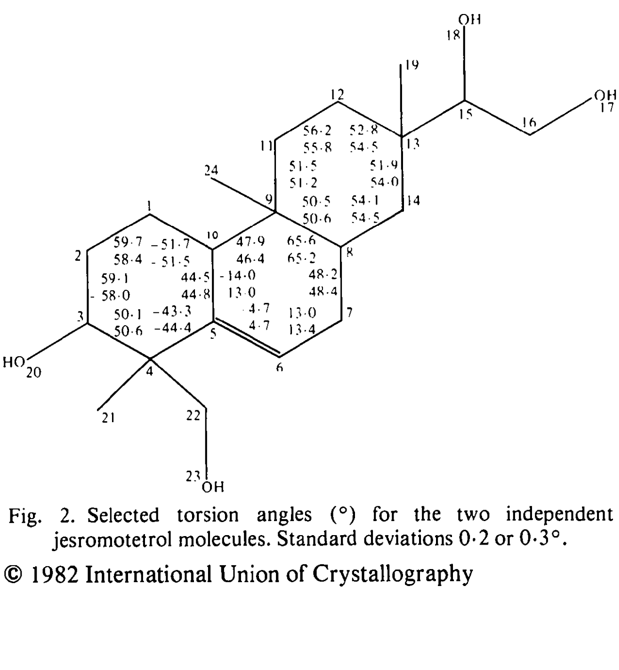

Fig. 2. Selected torsion angles (°) for the two independent jesromotetrol molecules. Standard deviations 0·2 or 0·3°.

* To whom correspondence should be addressed.

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Table 1. *Positional parameters* ($\times 10^4$, *for* H $\times 10^3$)

Molecule 1	x	y	z	U_{eq}/U^* ($Å^2$)	Molecule 2	x	y	z	U_{eq}/U^* ($Å^2$)
C(1)	4144 (1)	8300	5202 (2)	5·1 (2)	C(1)	9138 (1)	9097 (5)	8315 (2)	5·0 (2)
C(2)	3624 (1)	7269 (5)	4632 (2)	5·1 (2)	C(2)	8582 (1)	9848 (5)	7969 (2)	5·3 (2)
C(3)	3244 (1)	7610 (4)	5387 (2)	4·7 (2)	C(3)	8324 (1)	9273 (4)	6677 (2)	4·1 (2)
C(4)	3445 (1)	6791 (4)	6669 (2)	4·4 (2)	C(4)	8618 (1)	10113 (3)	5790 (2)	3·7 (2)
C(5)	3997 (1)	7514 (4)	7250 (2)	3·8 (2)	C(5)	9200 (1)	9617 (3)	6177 (2)	3·4 (2)
C(6)	4151 (1)	7987 (4)	8409 (2)	4·4 (2)	C(6)	9469 (1)	9114 (4)	5391 (2)	3·5 (2)
C(7)	4707 (1)	8352 (4)	9073 (2)	4·3 (2)	C(7)	10051 (1)	8961 (4)	5688 (2)	3·5 (2)
C(8)	5090 (1)	7662 (4)	8363 (2)	3·7 (2)	C(8)	10306 (1)	9907 (3)	6891 (2)	3·1 (2)
C(9)	4911 (1)	8489 (4)	7069 (2)	3·9 (2)	C(9)	10038 (1)	9147 (3)	7847 (2)	3·5 (2)
C(10)	4385 (1)	7463 (4)	6472 (2)	3·8 (2)	C(10)	9467 (1)	9937 (4)	7507 (2)	3·6 (2)
C(11)	5321 (1)	7870 (5)	6399 (2)	4·7 (2)	C(11)	10325 (1)	9999 (5)	9078 (2)	4·4 (2)
C(12)	5876 (1)	8407 (5)	7043 (2)	4·9 (2)	C(12)	10915 (1)	9643 (4)	9384 (2)	4·2 (2)
C(13)	6059 (1)	7539 (4)	8319 (2)	4·0 (2)	C(13)	11175 (1)	10483 (3)	8437 (2)	3·5 (2)
C(14)	5651 (1)	8170 (4)	9002 (2)	4·2 (2)	C(14)	10891 (1)	9584 (4)	7216 (2)	3·4 (2)
C(15)	6592 (1)	8491 (4)	8932 (2)	4·1 (2)	C(15)	11760 (1)	9952 (4)	8775 (2)	3·7 (2)
C(16)	6853 (1)	7525 (4)	10122 (3)	4·7 (2)	C(16)	11895 (1)	7732 (4)	8826 (3)	4·4 (2)
O(17)	7291 (1)	8705 (4)	10710 (2)	5·6 (2)	O(17)	12433 (1)	7565 (4)	9398 (2)	5·0 (2)
O(18)	6937 (1)	8340 (3)	8162 (2)	5·8 (2)	O(18)	12037 (1)	10909 (3)	8005 (2)	4·7 (2)
C(19)	6108 (1)	5235 (4)	8286 (3)	5·0 (2)	C(19)	11139 (1)	12793 (4)	8412 (2)	4·6 (2)
O(20)	3142 (1)	9722 (4)	5400 (2)	6·4 (2)	O(20)	8305 (1)	7104 (3)	6590 (2)	5·7 (2)
C(21)	3455 (1)	4457 (5)	6612 (4)	6·5 (3)	C(21)	8556 (1)	12431 (4)	5733 (3)	5·4 (2)
C(22)	3050 (1)	7278 (5)	7404 (3)	5·6 (2)	C(22)	8343 (1)	9381 (4)	4524 (2)	4·7 (2)
O(23)	2986 (1)	9374 (4)	7542 (2)	6·4 (2)	O(23)	8374 (1)	7261 (4)	4351 (2)	5·7 (2)
C(24)	4857 (1)	10790 (4)	7071 (3)	5·0 (2)	C(24)	10046 (1)	6823 (4)	7890 (2)	4·5 (2)
O(25)†	7404 (1)	12003 (4)	8131 (2)	7·3 (2)					
O(26)†	7377 (1)	5689 (4)	6896 (2)	6·0 (2)					
H(1*a*)	438 (2)	801 (8)	472 (4)	7·6 (11)	H(1*a*)	927 (2)	932 (8)	917 (4)	8·2 (12)
H(1*b*)	410 (2)	987 (8)	519 (4)	7·3 (11)	H(1*b*)	913 (2)	745 (8)	836 (4)	8·2 (12)
H(2*a*)	347 (2)	786 (10)	383 (5)	10·2 (15)	H(2*a*)	837 (2)	918 (10)	842 (5)	10·0 (15)
H(2*b*)	361 (2)	567 (8)	445 (4)	7·6 (11)	H(2*b*)	862 (1)	1141 (7)	800 (3)	6·9 (10)
H(3)	291 (1)	697 (6)	505 (3)	6·1 (9)	H(3)	796 (1)	984 (5)	642 (3)	4·6 (8)
H(6)	389 (1)	809 (6)	886 (3)	5·4 (8)	H(6)	933 (1)	881 (6)	462 (3)	5·5 (8)
H(7*a*)	477 (1)	742 (6)	989 (3)	5·2 (7)	H(7*a*)	1022 (2)	966 (10)	502 (5)	10·4 (16)
H(7*b*)	472 (1)	969 (5)	927 (3)	4·3 (7)	H(7*b*)	1020 (1)	754 (6)	570 (3)	5·9 (9)
H(8)	501 (2)	600 (8)	828 (4)	8·3 (12)	H(8)	1024 (1)	1139 (6)	679 (3)	5·5 (8)
H(10)	449 (2)	600 (7)	645 (3)	6·9 (10)	H(10)	950 (1)	1152 (6)	763 (3)	5·8 (8)
H(11*a*)	530 (1)	854 (7)	562 (4)	6·5 (9)	H(11*a*)	1022 (1)	1162 (7)	910 (3)	6·4 (9)
H(11*b*)	527 (1)	628 (6)	618 (3)	5·9 (9)	H(11*b*)	1017 (1)	948 (6)	972 (3)	5·4 (8)
H(12*a*)	590 (1)	988 (6)	716 (3)	5·6 (8)	H(12*a*)	1108 (1)	1039 (6)	1011 (3)	5·8 (8)
H(12*b*)	614 (2)	791 (10)	658 (5)	10·4 (15)	H(12*b*)	1101 (2)	818 (7)	949 (3)	6·9 (10)
H(14*a*)	568 (2)	740 (8)	978 (3)	7·5 (11)	H(14*a*)	1104 (1)	1023 (6)	661 (3)	5·9 (8)
H(14*b*)	570 (1)	956 (6)	911 (3)	5·7 (8)	H(14*b*)	1094 (1)	826 (6)	722 (3)	5·0 (8)
H(15)	654 (1)	995 (5)	906 (2)	4·0 (7)	H(15)	1192 (1)	1059 (5)	959 (3)	4·4 (7)
H(16*a*)	660 (2)	733 (7)	1049 (3)	6·5 (9)	H(16*a*)	1167 (1)	698 (5)	912 (3)	4·5 (7)
H(16*b*)	698 (1)	615 (7)	1000 (3)	6·1 (9)	H(16*b*)	1184 (1)	726 (6)	805 (3)	5·4 (8)
H(17)	741 (2)	806 (12)	1117 (6)	11 (2)	H(17)	1242 (2)	660 (9)	933 (4)	7·1 (15)
H(18)	707 (1)	914 (5)	816 (3)	4·8 (7)	H(18)	1186 (2)	1128 (1)	732 (5)	11·6 (17)
H(19*a*)	647 (2)	473 (7)	814 (3)	7·5 (11)	H(19*a*)	1123 (2)	1343 (8)	915 (4)	7·7 (11)
H(19*b*)	614 (1)	459 (6)	910 (3)	5·2 (8)	H(19*b*)	1076 (1)	1325 (6)	828 (3)	5·8 (8)
H(19*c*)	578 (2)	469 (9)	778 (4)	9·1 (13)	H(19*c*)	1126 (1)	1350 (6)	777 (3)	6·4 (9)
H(21*a*)	353 (2)	385 (8)	738 (4)	8·2 (12)	H(21*a*)	866 (2)	1300 (7)	647 (4)	6·4 (10)
H(21*b*)	366 (2)	398 (11)	620 (5)	11·5 (17)	H(21*b*)	864 (2)	1304 (8)	505 (4)	7·4 (11)
H(21*c*)	302 (2)	406 (10)	603 (5)	10·4 (15)	H(21*c*)	826 (2)	1284 (6)	562 (3)	5·6 (9)
H(22*a*)	318 (2)	671 (7)	819 (3)	7·1 (10)	H(22*a*)	849 (1)	1021 (5)	391 (3)	4·4 (7)
H(22*b*)	267 (2)	666 (8)	696 (4)	7·8 (11)	H(22*b*)	800 (1)	986 (6)	433 (3)	5·7 (8)
H(23)	275 (3)	955 (11)	776 (5)	10·5 (18)	H(23)	831 (2)	674 (11)	503 (5)	10·2 (16)
H(24*a*)	515 (1)	1140 (6)	763 (3)	6·1 (9)	H(24*a*)	998 (2)	635 (7)	868 (4)	7·0 (10)
H(24*b*)	479 (1)	1129 (7)	626 (4)	6·7 (10)	H(24*b*)	1046 (2)	641 (9)	800 (4)	8·6 (12)
H(24*c*)	459 (2)	1109 (8)	731 (4)	8·2 (13)	H(24*c*)	976 (1)	626 (6)	717 (3)	5·3 (8)

* $U_{eq} = (U_{11} U_{22} U_{33})^{1/3}$.
† Water of hydration.

significant decrease in intensity was observed. Lorentz and polarization corrections were applied, but no absorption corrections were made. The direct methods program *MULTAN* (Germain, Main & Woolfson, 1971) was used to calculate phases for the 400 $|E|$ values greater than 1·43. The phase set with the highest combined figure of merit was selected, and the E map calculated with these phases revealed the positions of two 22-atom fragments 34 atoms of which were used in the starting refinement set. Alternate least-squares refinement and difference-Fourier calculations yielded the coordinates of the missing 16 heavy atoms and all hydrogen atoms except for the waters of hydration. Least-squares refinement yielded a final R of 0·048, where $R = \sum||F_o| - |F_c||/\sum|F_o|$. The function minimized in the least-squares refinement was $\sum w(|F_o| - |F_c|)^2$, where $w = 1/\sigma(F_o)^2$ was determined from counting statistics. H-atom thermal parameters were refined isotropically.

A final difference map showed no peak larger than 0·3 e Å^{-3}. Atomic scattering factors and the real and imaginary contributions to the anomalous dispersion were calculated by the XRAY program (Stewart, Machin, Dickinson, Ammon, Heck & Flack, 1976). Atomic positional parameters are given in Table 1 while interatomic distances and torsion angles are shown in Figs. 1 and 2. Valence angles are listed in Table 2.*

Table 2. *Valence angles (standard deviation* 0·1–0·2°)

	Moelcule 1	Molecule 2
C(2)C(1)C(10)	111·0	112·7
C(1)C(2)C(3)	110·2	110·1
C(2)C(3)C(4)	112·5	112·6
C(2)C(3)O(20)	108·0	108·4
C(4)C(3)O(20)	110·8	109·2
C(3)C(4)C(5)	111·9	111·7
C(3)C(4)C(21)	108·4	108·8
C(3)C(4)C(22)	109·7	108·8
C(5)C(4)C(21)	107·9	108·3
C(5)C(4)C(22)	113·7	114·7
C(21)C(4)C(22)	104·7	104·7
C(4)C(5)C(6)	122·1	122·0
C(4)C(5)C(10)	116·6	116·0
C(6)C(5)C(10)	121·0	121·8
C(5)C(6)C(7)	124·3	124·1
C(6)C(7)C(8)	112·1	112·1
C(7)C(8)C(9)	109·3	110·0
C(7)C(8)C(14)	111·9	111·1
C(9)C(8)C(14)	114·4	113·8
C(8)C(9)C(10)	107·2	107·7
C(8)C(9)C(11)	108·0	109·4
C(8)C(9)C(24)	110·9	110·2
C(10)C(9)C(11)	109·2	109·3
C(10)C(9)C(24)	111·4	110·5
C(11)C(9)C(24)	110·1	109·7
C(1)C(10)C(5)	111·9	111·7
C(1)C(10)C(9)	112·0	113·0
C(5)C(10)C(9)	113·0	112·3
C(9)C(11)C(12)	114·3	113·2
C(11)C(12)C(13)	114·4	113·2
C(12)C(13)C(14)	107·2	108·0
C(12)C(13)C(15)	108·4	109·9
C(12)C(13)C(19)	110·8	109·7
C(14)C(13)C(15)	109·3	111·8
C(14)C(13)C(19)	110·8	110·8
C(15)C(13)C(19)	110·1	106·6
C(8)C(14)C(13)	114·6	113·9
C(13)C(15)C(16)	113·5	116·5
C(13)C(15)O(18)	109·9	112·4
C(16)C(15)O(18)	108·5	107·4
C(15)C(16)O(17)	109·4	107·2
C(4)C(22)O(23)	112·7	114·5

Discussion. Fig. 3 is an *ORTEP* drawing (Johnson, 1965) of jesromotetrol. The flattened chair, half-chair and chair conformations of the three six-membered rings reflect the influence of the C(5)–C(6) double bond. Two independent tetrahydroxy and two independent water molecules per asymmetric unit provide numerous opportunities for hydrogen-bond formation. Since hydrogen atoms associated with the

* Lists of structure factors and anisotropic parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 36186 (26 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

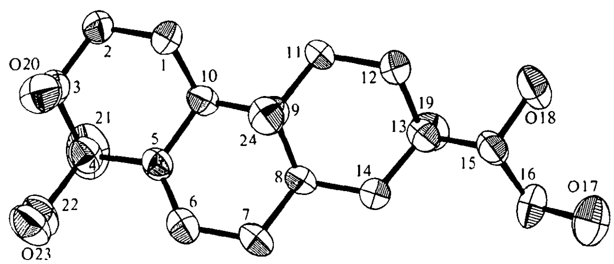

Fig. 3. *ORTEP* (Johnson, 1965) drawing of jesromotetrol. Thermal ellipsoids are drawn at the 35% probability level.

Table 3. *Inter- and intramolecular oxygen–oxygen separations*

Atom pair	d (Å)	Transformation of second atom
O(20)–O(23)	2·620 (4)	(x,y,z)
O(20)′–O(23)′*	2·641 (3)	(x,y,z)
O(17)–O(18)	2·864 (4)	(x,y,z)
O(17)′–O(18)′	2·782 (3)	(x,y,z)
H_2O(26)–O(18)	2·729 (3)	(x,y,z)
H_2O(25)–O(18)	2·728 (4)	(x,y,z)
O(20)′–H_2O(26)	2·735 (3)	(x,y,z)
O(18)′–O(23)	2·883 (3)	$(1 + x, y, z)$
H_2O(25)–H_2O(26)	2·819 (4)	$(x, 1 + y, z)$
O(18)′–O(17)	2·731 (3)	$(2 - x, \frac{1}{2} + y, 2 - z)$
O(17)–O(17)′	2·670 (4)	$(2 - x, \frac{1}{2} + y, 2 - z)$
H_2O(25)–O(17)′	2·801 (3)	$(2 - x, \frac{1}{2} + y, 2 - z)$
O(18)′–O(23)′	2·809 (3)	$(2 - x, \frac{1}{2} + y, 1 - z)$
O(20)–H_2O(26)	2·736 (3)	$(1 - x, \frac{1}{2} + y, 1 - z)$

* ′ refers to molecule 2 in the table of atomic coordinates.

water molecules were not located, the hydrogen-bonding network cannot be explicitly described; however, Table 3 lists inter- and intramolecular oxygen–oxygen distances of less than 3·00 Å.

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The Ion Radical Salt of 1-Methyl-3-propylimidazolium with 7,7,8,8-Tetracyano-*p*-quinodimethane: MPI$^+$.TCNQ$^{\cdot-}$

BY V. LANGER AND K. HUML

Institute of Macromolecular Chemistry, Czechoslovak Academy of Sciences, 162 06 *Praha* 6, *Czechoslovakia*

AND G. RECK

Zentralinstitut für Molekularbiologie, Akademie der Wissenschaften der DDR, Berlin-Buch, German Democratic Republic

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Abstract. $C_7H_{13}N_2^+.C_{12}H_4N_4^{\cdot-}$, M_r = 329·39, triclinic, $P\bar{1}$, a = 10·982 (2), b = 14·337 (2), c = 8·750 (1) Å, α = 91·37 (1), β = 139·09 (1), γ = 94·40 (1)°, V = 892·3 (2) Å^3 at 295 K, Z = 2, F(000) = 346, D_x = 1·226 (1), D_m = 1·211 (1) Mg m^{-3} (by flotation), graphite-monochromated Cu $K\alpha$ radiation, μ = 0·573 mm^{-1}. The structure was solved by combined direct and Patterson methods and refined to R = 0·042 for 2464 counter reflexions. The interplanar distance between TCNQ anions is 3·13 (1) Å.

Introduction. The family of tetracyanoquinodimethane (TCNQ) salts and charge-transfer complexes is of interest, as several of the best-known one-dimensional organic metals are its members. Although the ion radical salt of 1-methyl-3-propylimidazolium (MPI) and TCNQ with stoichiometry 1:1 is not a good conductor (σ_{300K} = 3·1 × 10^{-5} Ω^{-1} m^{-1} for a powder sample; Šorm, Nešpůrek, Procházka & Koropecký, 1982), the arrangement of TCNQ anions relative to that of MPI cations is worthy of study.

The molecular formula of the title compound was confirmed by elemental analysis. The cell parameters and intensities (θ–2θ scan) were measured on a Syntex $P2_1$ automated diffractometer (Cu $K\alpha$) under conditions described by Langer & Huml (1978). In the range up to 2θ = 127°, 2941 independent reflexions were measured, 2465 of which were observed (I > 1·96σ_I). The data were corrected for the Lorentz–polarization factor (for formula see Langer, Huml & Zachová, 1979), but not for absorption.

The first attempt to solve the structure was made using *MULTAN* 78 (Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978), but a chicken-wire-like structure resulted, where the TCNQ anion might be placed in seven overlying positions, although the orientation was still the same. As the E statistics indicated a centrosymmetric structure, the Patterson function was calculated and the highest peak (origin removed) was regarded as an overlap of 16 parallel vectors between centrosymmetrically related TCNQ anions and, therefore, the translation vector for TCNQ

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