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X-RAY CRYSTAL STRUCTURE OF RACEMIC BIS(ETHYL FUMARATE)(ACETONITRILE)NICKEL(0)

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Summary

The crystal and molecular structure of racemic bis(ethyl fumarate)(acetonitrile)nickel(0) was determined from X-ray diffractometer data. The structure was solved by Patterson and Fourier methods, and refined by least-squares techniques to $R = 0.045$ for 3034 independent reflections.

Crystals are triclinic, space group $P\bar{1}$, with $Z = 2$, in a unit cell of dimensions: a 9.219 (6), b 13.257 (3), c 10.521 (9) Å, α 92.57 (9)°, β 109.25 (10)°, γ 110.0 (9)°.

The coordination around the nickel(0) atom (the four carbon atoms of the —HC=CH— groups, and the N atom of the acetonitrile residue), may be described as planar, with a slightly distorted trigonal symmetry. The conformations of the two ethyl fumarate residues agree, more or less closely, with the principle of staggered bonds. The torsion angles around the —HC=CH— groups are significantly different from 180°, as observed in other similar complexes. The four asymmetric carbon atoms of a molecule have the same absolute configuration. *SSSS* and *RRRR* molecules are present in the unit cell.

Introduction

Some Ni(0) organometallic compounds containing R—C≡N ligands, where R is an alkyl or an aryl radical, have been recently synthesized by Guerrieri and Salerno [1]. The structure of these compounds is of interest both for the study of the coordination around the metal and for understanding the way in which the nitrile ligand is bonded to the metal atom.

The crystal structure of racemic bis(ethyl fumarate)(acetonitrile)nickel(0), (I), is now reported.

Experimental

Preliminary space group and unit cell dimensions were obtained from Weissenberg photographs.

A crystal, essentially a parallepiped having dimensions ca. 0.40 × 0.30 × 0.50 mm, was mounted on a Picker FACS 1 four-circle computer-controlled diffractometer, equipped with a scintillation counter and a pulse-height analyser.

The orientation matrix, and cell dimensions, were obtained from a least-squares fit of χ, ϕ, ω and 2θ values from 12 independent reflections.

Crystal data

$C_{18}H_{27}NNiO_8$, $M = 444.13$. Triclinic: a 9.219 (6), b 13.257 (3), c 10.521 (9) Å, α 92.57 (9)°, β 109.25 (10)°, γ 110.0 (9)°, U 1113.77 Å^3, D_m 1.30, Z 2, D_c 1.32.

Space group $P\bar{1}$. Cu-Kα radiation, λ 1.54178 Å, μ(Cu-Kα) 15.6 cm^{-1}, μ(Mo-Kα) 8.9 cm^{-1}.

The above crystal was then mounted on a Philips PW 1100 computer-controlled diffractometer.

Intensity data were collected by use of graphite monochromatized Mo-Kα radiation with $2\theta \leqslant 50°$. The moving-crystal stationary counter technique was used, with ω-scan rate of 2° min^{-1} and a scan range of 1.50°. Background counts of 10 s duration were measured at each end of every ω scan. Three standard reflections were measured after every 120 reflections. The recentering of the crystal, when necessary, was automatically performed. 3912 independent reflections were measured, of which 3034 were considered observed and had $I \geqslant 2.5\ \sigma$. An arbitrary intensity equal to 0.5, the observable limit, was assigned to the non-significant reflections. All intensities were corrected for Lorentz and polarization effects. No absorption correction was introduced.

Determination and refinement of the structure

The structure was solved by the heavy atom method. A three-dimensional Patterson map permitted the location of the Ni atoms in the unit cell. All the non-hydrogen atoms have been localized by means of successive Fourier maps.

The positional and isotropic thermal parameters of the non-hydrogen atoms were refined by some cycles of block-diagonal least-squares, by use of a program of Immirzi [2]. Atomic scattering factors were calculated from the expression given in ref. 3, using values for the parameters given in ref. 4. The weighting scheme of ref. 5 was adopted:

$$\frac{1}{W} = A + B|F_0| + C|F_0|^2$$

where $A = 2/F_0$ (min), $B = 1.00$, and $C = 2/F_0$ (max). At this point the conventional R was 0.110 for the 3034 non-zero reflections, and the total R was 0.132.

Eight cycles of block-diagonal refinement were run, assuming anisotropic thermal parameters for all non-hydrogen atoms. The hydrogen atoms were also

TABLE 1

FINAL FRACTIONAL COORDINATES OF THE BIS(ETHYL FUMARATE)(ACETONITRILE)-NICKEL MOLECULE

Estimated standard deviations in parentheses.

Atom	x/a	y/b	z/c	B (Å^2)
Ni	0.44325(5)	0.17601(4)	0.29853(4)	
O(1)	0.8469(4)	0.2631(3)	0.5907(3)	
O(2)	0.6254(4)	0.2645(3)	0.6318(3)	
O(3)	0.5920(5)	0.4049(3)	0.1860(4)	
O(4)	0.4306(4)	0.4614(3)	0.2645(4)	
O(5)	0.1228(3)	0.2816(2)	0.2832(3)	
O(6)	0.1052(4)	0.1282(3)	0.3697(3)	
O(7)	0.2688(4)	0.0710(3)	−0.0347(3)	
O(8)	0.2398(4)	−0.0803(2)	0.0566(3)	
N	0.5484(4)	0.0760(3)	0.3227(3)	
C(1)	1.0620(9)	0.2150(8)	0.7344(7)	
C(2)	0.9102(8)	0.2350(5)	0.7248(5)	
C(3)	0.7019(5)	0.2745(3)	0.5567(4)	
C(4)	0.6525(5)	0.3009(3)	0.4191(4)	
C(5)	0.5223(5)	0.3362(3)	0.3732(4)	
C(6)	0.5189(5)	0.4017(3)	0.2630(5)	
C(7)	0.4122(8)	0.5292(5)	0.1581(7)	
C(8)	0.2522(12)	0.4792(7)	0.0566(8)	
C(9)	0.0828(12)	0.4297(6)	0.3760(12)	
C(10)	0.0715(7)	0.3194(5)	0.3861(6)	
C(11)	0.1409(5)	0.1862(3)	0.2910(4)	
C(12)	0.2118(4)	0.1638(3)	0.1914(4)	
C(13)	0.2217(5)	0.0622(3)	0.1744(4)	
C(14)	0.2451(5)	0.0221(3)	0.0539(4)	
C(15)	0.2795(6)	−0.1271(4)	−0.0491(5)	
C(16)	0.2781(9)	−0.2366(5)	−0.0167(6)	
C(17)	0.6147(5)	0.0183(3)	0.3359(4)	
C(18)	0.7024(6)	−0.0552(4)	0.3504(5)	
H(2)	0.942	0.302	0.806	5.00
H(21)	0.815	0.162	0.737	5.00
H(4)	0.712	0.291	0.349	5.00
H(5)	0.428	0.317	0.418	5.00
H(7)	0.427	0.611	0.206	5.00
H(71)	0.511	0.545	0.118	5.00
H(10)	0.156	0.319	0.489	5.00
H(101)	−0.054	0.263	0.374	5.00
H(120)	0.253	0.224	0.131	5.00
H(13)	0.216	0.013	0.253	5.00
H(15)	0.401	−0.076	−0.047	5.00
H(151)	0.186	−0.138	−0.149	5.00
H(18)	0.739	−0.070	0.453	5.00
H(181)	0.812	−0.020	0.324	5.00
H(182)	0.620	−0.134	0.281	5.00

introduced into the calculations but not refined, their coordinates being defined on stereochemical grounds and according to a difference-Fourier synthesis. The hydrogen atoms pertaining to the methyl groups of the C_2H_5 terminal groups were not introduced into the calculations, because of the very high thermal motions of the corresponding carbon atoms. The refinement converged to R = 0.045, while the total R was 0.065. The final shifts of the atomic parameters were negligible; all being well below the corresponding σ.

Table 1 reports the final fractional coordinates and the corresponding estimat-

TABLE 2

ANISOTROPIC THERMAL PARAMETERS [a] FOR THE NON-HYDROGEN ATOMS

Atom	B_{11}	B_{22}	B_{33}	B_{12}	B_{13}	B_{23}
Ni	3.59(2)	3.81(2)	3.69(2)	2.16(2)	1.67(1)	1.04(1)
O(1)	4.92(13)	7.31(16)	4.90(13)	3.34(12)	1.94(11)	2.10(12)
O(2)	6.83(17)	8.30(19)	5.26(14)	3.77(15)	3.52(13)	1.89(13)
O(3)	10.70(25)	8.32(20)	9.10(21)	6.49(20)	2.24(20)	5.05(18)
O(4)	8.00(19)	7.44(18)	10.55(23)	5.63(17)	6.16(18)	5.60(17)
O(5)	5.14(13)	5.67(14)	6.43(15)	2.98(11)	3.30(12)	0.92(11)
O(6)	6.79(17)	7.87(18)	6.25(15)	3.80(15)	4.09(14)	2.79(14)
O(7)	8.03(19)	6.66(16)	5.03(14)	4.02(15)	3.00(13)	1.73(12)
O(8)	6.22(15)	4.62(12)	5.29(13)	2.54(11)	2.52(11)	0.42(10)
N	4.69(14)	4.77(14)	4.58(14)	2.80(12)	1.48(11)	0.84(11)
C(1)	9.72(42)	17.12(68)	8.93(38)	9.63(48)	2.85(32)	5.54(42)
C(2)	8.53(32)	9.91(36)	5.80(24)	5.25(29)	2.52(23)	3.45(24)
C(3)	4.58(17)	4.33(16)	4.76(17)	1.87(14)	2.13(14)	0.74(13)
C(4)	4.12(15)	4.46(16)	4.60(16)	2.02(13)	2.05(13)	1.00(13)
C(5)	4.15(15)	3.93(15)	5.19(17)	1.90(13)	2.43(13)	1.10(13)
C(6)	5.21(19)	4.61(17)	6.74(22)	2.57(15)	3.42(17)	2.16(16)
C(7)	9.96(38)	8.64(34)	11.51(42)	6.14(31)	6.16(34)	6.64(33)
C(8)	13.11(59)	12.09(56)	9.43(45)	10.04(46)	2.15(42)	5.69(42)
C(9)	15.23(67)	6.62(34)	23.64(96)	1.51(37)	14.07(72)	—2.55(45)
C(10)	7.13(28)	8.16(30)	9.34(33)	3.54(25)	5.19(26)	0.07(25)
C(11)	3.85(15)	5.71(19)	4.78(17)	2.49(14)	1.93(13)	1.23(14)
C(12)	3.75(15)	4.74(16)	4.43(15)	2.24(13)	1.87(12)	1.13(13)
C(13)	4.28(16)	4.46(16)	4.12(15)	2.05(13)	1.52(13)	0.93(13)
C(14)	3.96(15)	4.73(17)	4.31(16)	1.94(13)	1.48(13)	0.88(13)
C(15)	5.37(20)	5.71(21)	5.47(20)	2.05(17)	1.86(16)	—0.85(16)
C(16)	10.93(41)	7.41(30)	8.31(32)	5.59(30)	3.80(30)	0.33(25)
C(17)	4.68(17)	5.01(17)	4.60(17)	2.76(15)	1.80(14)	1.38(14)
C(18)	6.32(23)	6.27(22)	7.44(25)	4.55(20)	3.07(20)	2.71(19)

[a] The temperature factor is in the form: $T_j = \exp[-\frac{1}{4}(B_{11}a^{*2}h^2 + B_{22}b^{*2}k^2 + B_{33}c^{*2}l^2 + 2B_{12}a^*b^*hk + 2B_{13}a^*c^*hl + 2B_{23}b^*c^*kl)]$

ed standard deviations of the atoms of the molecule. Table 2 lists the anisotropic thermal parameters of the non-hydrogen atoms. Calculated and observed structure amplitudes are listed in a table that may be obtained by application to the Authors.

Results and discussion

Views of the molecule of racemic bis(ethyl fumarate)acetonitrilenickel(0) are shown in Figs. 1 and 2. Both figures have been obtained by the ORTEP computing and drawing programs [6]. The geometric parameters of the molecule, with their estimated standard deviations, are reported in Table 3.

The nickel atom is bonded to five atoms; the two carbon atoms of the central CH=CH groups of both ethyl fumarate molecules, and to the nitrogen atom of the acetonitrile residue. The five-fold coordination of the nickel atom is shown in Fig. 1. The average Ni—C distance is 2.005 (5) Å, while the Ni—N distance is 1.888 (4) Å. These values are in good agreement with the data reported for many similar compounds [7—14].

The atoms Ni, C(4), C(5), C(12), C(13) and N are nearly coplanar, 0.019 Å being the r.m.s. distance of an atom from the least-squares plane. The four

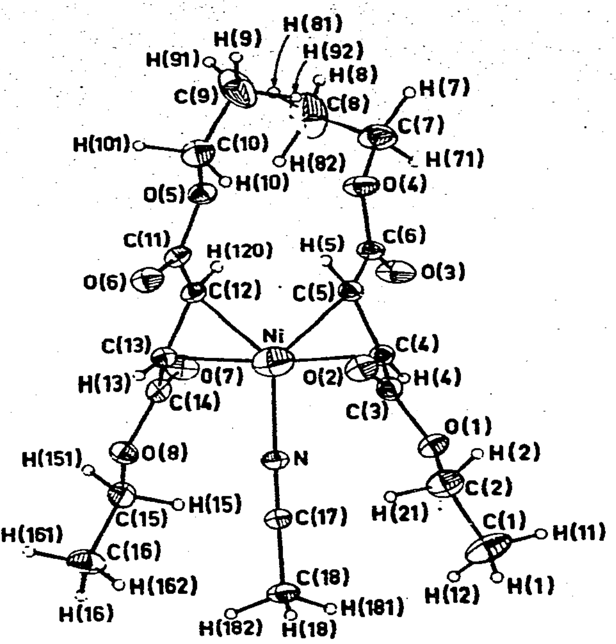


Fig. 1. View of the bis(ethyl fumarate)(acetonitrile)nickel molecule indicating the atom labelling scheme, and the 30% probability thermal vibration ellipsoids (*RRRR* stereoisomer).

hydrogen atoms, H(4), H(5), H(120), and H(13), the location of which was confirmed by a difference-Fourier map, are displaced by about 1 Å from this plane; two below, H(4) and H(120), and two above, H(5) and H(13). The Ni···H separation is about 2.4 Å. By assuming that the Ni—C bonds may be directed along the straight line joining the Ni atom and the mid point of the two HC=CH groups, the geometry of the coordination at the Ni atom is roughly trigonal. The N—Ni—C_{Mid} bond angles are 112.95° and 113.8°, while the C_{Mid}—Ni—C_{Mid} bond angle is 133.25°. These distortions with respect to a

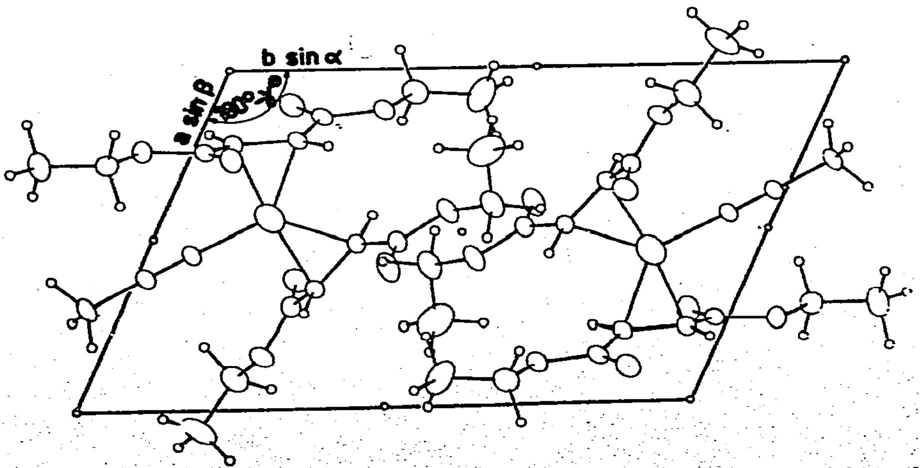


Fig. 2. Packing arrangement of bis(ethyl fumarate)(acetonitrile)nickel as viewed down the c axis.

TABLE 3

MOST SIGNIFICANT GEOMETRIC PARAMETERS OF THE MOLECULE OF BIS(ETHYL FUMARATE)-(ACETONITRILE)NICKEL(0)

(a) Bond lengths (Å)	
Ni—C(4)	1.999(5)
Ni—C(5)	2.009(4)
Ni—C(12)	2.004(5)
Ni—C(13)	2.006(5)
Ni—N	1.888(4)
C(1)—C(2)	1.49(1)
C(2)—O(1)	1.46(1)
O(1)—C(3)	1.34(1)
C(3)—O(2)	1.21(1)
C(3)—C(4)	1.47(1)
C(4)—C(5)	1.39(1)
C(5)—C(6)	1.48(1)
C(6)—O(3)	1.21(1)
C(6)—O(4)	1.33(1)
O(4)—C(7)	1.47(1)
C(7)—C(8)	1.40(2)
C(9)—C(10)	1.44(1)
C(10)—O(5)	1.46(1)
O(5)—C(11)	1.34(1)
O(6)—C(11)	1.20(1)
C(11)—C(12)	1.48(1)
C(12)—C(13)	1.39(1)
C(13)—C(14)	1.47(1)
O(7)—C(14)	1.20(1)
C(14)—O(8)	1.35(1)
O(8)—C(15)	1.46(1)
C(15)—C(16)	1.50(1)
N—C(17)	1.129(6)
C(17)—C(18)	1.46(1)

(b) Bond angles (deg.)	
C(4)—Ni—C(5)	40.7(3)
C(4)—Ni—N	92.6(1)
N—Ni—C(13)	93.5(1)
C(12)—Ni—C(13)	40.6(3)
C(5)—Ni—C(12)	92.6(1)
N—Ni—C(5)	133.3(1)
N—Ni—C(12)	134.1(1)
Ni—N—C(17)	178(8)
N—C(17)—C(18)	178(7)
Ni—C(4)—C(5)	70.1(2)
Ni—C(5)—C(4)	69.3(2)
Ni—C(12)—C(13)	69.8(2)
Ni—C(13)—C(12)	69.6(2)
C(1)—C(2)—O(1)	107.4(3)
C(2)—O(1)—C(3)	116.8(2)
O(1)—C(3)—O(2)	123.0(2)
O(1)—C(3)—C(4)	110.9(2)
O(2)—C(3)—C(4)	126.2(2)
C(3)—C(4)—C(5)	120.2(2)
C(4)—C(5)—C(6)	117.9(2)
C(5)—C(6)—O(4)	110.5(2)
C(5)—C(6)—O(3)	125.2(2)
O(3)—C(6)—O(4)	124.2(2)
C(6)—O(4)—C(7)	116.8(3)
O(4)—C(7)—C(8)	109.6(4)
C(9)—C(10)—O(5)	108.8(3)
C(10)—O(5)—C(11)	116.2(2)
O(5)—C(11)—C(12)	110.7(2)
O(5)—C(11)—O(6)	123.9(2)
O(6)—C(11)—C(12)	125.4(2)
C(11)—C(12)—C(13)	118.5(2)
C(12)—C(13)—C(14)	121.3(2)
C(13)—C(14)—O(7)	126.9(2)
C(13)—C(14)—O(8)	110.1(2)
O(7)—C(14)—O(8)	123.1(2)
C(14)—O(8)—C(15)	116.5(2)
O(8)—C(15)—C(16)	105.9(2)

(c) Torsion angles (deg.)	
C(1)—C(2)—O(1)—C(3)	174
C(2)—O(1)—C(3)—C(4)	181
C(2)—O(1)—C(3)—O(2)	1
O(1)—C(3)—C(4)—C(5)	192
C(3)—C(4)—C(5)—C(6)	156
O(2)—C(3)—C(4)—C(5)	12
C(4)—C(5)—C(6)—O(4)	203
C(4)—C(5)—C(6)—O(3)	19
C(5)—C(6)—O(4)—C(7)	181
C(6)—O(4)—C(7)—C(8)	76
O(3)—C(6)—O(4)—C(7)	5
C(9)—C(10)—O(5)—C(11)	171
C(10)—O(5)—C(11)—C(12)	187

(d) Intramolecular distances between non-bonded atoms (Å)	
Ni···O	⩾3.30
Ni···C	⩾2.82
Ni···H	⩾2.40
N···C	⩾2.81
N···O	⩾3.20
O···O	⩾2.25
O···C	⩾3.50
C···C	⩾3.60

(continued)

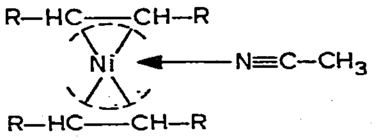
TABLE 3 (continued)

(c) Torsion angles (deg.)	
C(10)—O(5)—C(11)—O(6)	6
O(5)—C(11)—C(12)—C(13)	187
O(6)—C(11)—C(12)—C(13)	8
C(11)—C(12)—C(13)—C(14)	163
C(12)—C(13)—C(14)—O(8)	184
C(12)—C(13)—C(14)—O(7)	5
C(13)—C(14)—O(8)—C(15)	187
O(7)—C(14)—O(8)—C(15)	5
C(14)—O(8)—C(15)—C(16)	176
Ni—N—C(17)—C(18)	—25
N—Ni—C(13)—C(14)	—62
N—Ni—C(13)—C(12)	180
N—Ni—C(12)—C(11)	64
N—Ni—C(12)—C(13)	1
N—Ni—C(4)—C(3)	—62
N—Ni—C(4)—C(5)	182
N—Ni—C(5)—C(6)	65
N—Ni—C(5)—C(4)	2

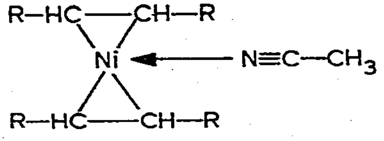
perfectly trigonal symmetry may derive from Van der Waals repulsions between atoms of the facing ethyl fumarate and acetonitrile residues. Similar trigonal symmetries have been quoted for tris(ethylene)nickel, for tris(bicycloheptene)-nickel(0) [15], for all *cis*-1,5,9-cyclododecatrienenickel(0) [16], and for bis-(t-butylisocyanide)(azobenzene)nickel(0) [17]. In I, the torsion angles on the Ni—C bonds, with respect to the Ni—N bond, are nearly 180°, 60° or 300°, while the C(13) and C(4) carbon atoms eclipse the nitrogen atom, the interatomic distances being 2.84 Å and 2.81 Å respectively.

A number of penta-coordinated complexes, in contrast to racemic bis(ethyl fumarate)(acetonitrile)nickel, have coordination polyhedra around the metal that may be described as trigonal bipyramids or as tetragonal pyramids [18].

In I the bonding situation around the metal may be represented by two limiting schemes:



(A)



(B)

In the limiting structure A, the two carbon atoms of the R—HC=CH—R groups have sp^2 hybridization, and the bonding to the nickel atom is via the delocalized π electrons. In the limiting structure B, the carbon atoms of the R—HC=CH—R groups are σ bonded to the nickel atom and, hence, are closer to sp^3 hybridization.

The nickel atom does not reach the stable electronic structure of Kr, and this fact may explain the great reactivity of the complex. The acetonitrile ligand is not unusual, the Ni, N, C(17) and C(18) atoms lying on an approximately straight line, with N—C(17) distance of 1.129 (6) Å, indicating a triple bond.

The structures of the two ethyl fumarate ligands agree, more or less closely, with the principle of staggered bonds. The torsion angles for the bonds of the backbone chains all correspond to a *trans* configuration (planar zig-zag conformations) with the exception of the O(4)—C(7) bond, at which the torsion angle is nearly *gauche* (76°). This *gauche* conformation may find its origin in packing requirements. The oxygen side atoms, as expected, nearly eclipse the carbon atoms three bonds away.

An interesting structural feature of the molecule I is the departure from coplanarity of the carbon atoms of both fumaric acid residues. In fact the C(3)—C(4)—C(5)—C(6) internal rotation angle is 156°, while the C(11)—C(12)—C(13)—C(14) angle is 163°. These values are in line with the values quoted for racemic tetracarbonyl(fumaric acid)iron (145 ± 2°) [19], and for the A, B and C independent molecules of (—)tetracarbonyl(fumaric acid)iron (154.2, 153.1, 142.5°) [20]. In contrast the carbon atoms of free fumaric acid are strictly coplanar [21]. The reason for these distortions, present in molecule I, may be found in the necessity of relaxing intramolecular contacts between the nitrogen atom and C(3) and C(14). In I these distances are 3.079 (5) Å [N···C(3)] and 3.088 (6) Å [N···C(14)], but would be considerably shorter if the fumaric acid residues were planar.

According to the sequence rule proposed by Cahn, Ingold and Prelog [22], the asymmetric centres of the bis(ethyl fumarate)(acetonitrile)nickel(0) molecule, [C(4), C(5), C(12), and C(13)] have the same absolute configuration. The configuration of the four carbon atoms on the basis of the coordinates reported in Table 1 may be described as *SSSS* and the isolated molecule of I, therefore, may present optical activity. The crystals of I are optically inactive due to the presence of the symmetry centres in the unit cell.

All the intramolecular contact distances reach acceptable values (see Table 3). The intermolecular Van der Waals interactions are also acceptable, no C···O distance being $<$ 3.40 Å, while the shortest N···C, C···C and O···O distances are 3.70, 3.60 and 3.99 Å respectively.

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