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The Crystal Structure of Bis-(N-isopropyl-3-ethylsalicylaldiminato)palladium

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The crystal structure of bis-(*N*-isopropyl-3-ethylsalicylaldiminato)palladium, $Pd(C_{12}H_{16}NO)_2$, has been determined from three-dimensional data collected on a single-crystal diffractometer with Mo K α radiation. The cell has dimensions a=10.672, b=13.063, and c=7.998 Å, belongs to space group $P2_1/a$, and contains two molecules. The structure consists of discrete molecules in which palladium(II) has strictly planar coordination configuration. The molecular structure shows the steric factors which cause the tetrahedral configuration to appear for the corresponding nickel(II) chelate.

Introduction

It has been shown (Holm & Swaminathan, 1963) that 3-substituted bis-(N-isopropylsalicylaldiminato)nickel chelates exist in a conformational equilibrium between a planar species and a tetrahedral species in toluene solution. In the crystalline state, however, these chelates exhibit a striking alternation in coordination configuration as the 3-substituent is changed from hydrogen to methyl to ethyl. That is, consistent with the magnetic moments reported by Holm & Swaminathan (1963), the coordination configurations have been shown to be tetrahedral for the 3-hydrogen chelate (Fox, Orioli, Lingafelter & Sacconi, 1964), planar for the 3-methyl chelate (3-M-Ni: Braun & Lingafelter, 1966), and tetrahedral for the 3-ethyl chelate (3-E-Ni: Braun & Lingafelter, 1967). The molecular structure of the planar chelate 3-M-Ni has shown the steric factors which cause the tetrahedral configuration to appear for N-isopropylsalicylaldimine chelates. In order to examine the intramolecular steric interactions of the planar 3-ethyl chelate, we have now completed the crystal structure determination of bis-(N-isopropyl-3ethylsalicylaldiminato)palladium (3-E-Pd) by threedimensional X-ray diffraction techniques.

Experimental

Bis-(*N*-isopropyl-3-ethylsalicylaldiminato)palladium was prepared by the method of Sacconi, Paoletti & Del Re (1957), using 3-ethylsalicylaldehyde which was prepared according to the general procedure of the Duff (1941) reaction. Final purification was accomplished by two recrystallizations from equal volumes of chloroform and ligroin by slow evaporation at room temperature. The crystal used for determination of cell dimensions and collection of intensity data was a well-defined rhomb, tabular on (001) and bounded by $\{011\}$, approximate dimensions $0.25 \times 0.20 \times 0.12$ mm.

Cell dimensions were determined on a Picker X-ray diffractometer equipped with a General Electric goniostat, using Mo $K\alpha$ radiation ($\lambda = 0.71069$ Å). The cell dimensions with their standard deviations are

$a = 10.672 \pm 0.002$	Å
$b = 13.063 \pm 0.002$	
$c = 7.998 \pm 0.001$	
$\beta = 98.09 \pm 0.01^{\circ}$	

Systematic absences of 0k0 for k odd and h0l for h odd identified the space group as $P2_1/a$. The cell contains two molecules: measured density 1.47 g.cm⁻³, calculated density 1.465 g.cm⁻³.

The intensity data were collected by the ω -2 θ scan method (Furnas, 1957), using zirconium-filtered Mo K α radiation and a Picker diffractometer equipped with a scintillation counter, pulse-height discriminator, and a General Electric single-crystal goniostat. A suitable scan range for each reflection was calculated by the formula of Alexander & Smith (1964), scan range = $1.8 + 1.0(\tan \theta)$. Stationary background measurements were made at the start and finish of each scan. Of the 2061 reflections which were examined within the limiting Cu K α sphere, 1556 reflections had an intensity (I) greater than twice the standard deviation of the intensity ($2\sigma_I$). These 1556 reflections ranged in intensity from 1 to 18,000. The remaining 505 reflections were coded 'unobserved' and were assigned an intensity of $2\sigma_I$ for later least-squares refinement.

Four standard reflections were measured periodically to detect systematic changes during the course of the data collection. The scale factor determined by these standard reflections was essentially constant, with only a random variation of $\pm 0.5\%$ due primarily to counting statistics. These periodic scale factors were not applied.

Structure determination

All calculations were carried out on an IBM 7094 computer with a set of programs written or adapted by Stewart (1964). Lorentz and polarization factors were applied and relative structure factors were calculated without correction for absorption. The atomic scattering factors used were those of Thomas & Umeda (1957) for palladium, those of Berghuis, Haanappel, Potters, Loopstra, MacGillavry & Veenendaal (1955) for oxygen, nitrogen, and carbon, and those from Table II of Stewart, Davidson & Simpson (1965) for hydrogen. No correction was made for anomalous dispersion.

Since there are two molecules in the cell of space group $P2_1/a$, the palladium atoms are required to be at centers of symmetry. A three-dimensional Fourier synthesis was calculated, using phases determined by the palladium atoms at (0,0,0) and $(\frac{1}{2},\frac{1}{2},0)$. Such a Fourier synthesis shows mirror planes in (x,0,z) and $(x,\frac{1}{2},z)$. Another three-dimensional Fourier synthesis phased on the palladium atom and on a nitrogen atom arbitrarily selected from the mirror-related pair at 2 Å from the palladium atom destroyed the psuedo-mirror planes. This and a third Fourier synthesis enabled the positions of all 15 non-hydrogen atoms to be determined and resulted in a reduction of R to 0.15. R is defined throughout as $\Sigma ||F_0| - |F_c||/\Sigma |F_0|$, where the sums are over the unique, observed reflections only.

Further refinement was carried out with the fullmatrix least-squares program of Busing & Levy (1959) as adapted in the UW crystallographic calculation system (Stewart, 1964). The function minimized was $\Sigma w(|F_o|$ $-|F_c|^2$, where w=1 for observed reflections and for unobserved reflections for which $F_c > F_o$ and w = 0 for unobserved reflections for which $F_c \leq F_o$. Three cycles of least-squares refinement using individual isotropic temperature factors followed by one cycle using individual anisotropic temperature factors reduced R to 0.046. Introduction of all 16 hydrogen atoms, which had been clearly located from a three-dimensional ΔF synthesis, reduced R to 0.039. Two more cycles of refinement of the non-hydrogen atoms with anisotropic temperature factors and four cycles of refinement of the hydrogen atoms with individual isotropic temperature factors reduced R to 0.024. During these and all subsequent least-squares calculations, the calculated structure factor contributions of the parameters not being refined were included as fixed contributions.

Considering the root-mean-square ΔF as an average standard deviation, a weighting scheme for further

Table 2. Parameters of hydrogen atoms
and their estimated standard deviations

	Position x/a	al parameter y/b	$\times 10^{3}$ z/c	I hermal parameter B
4)	- 226 (3)	87 (2)	657 (4)	4 (1)
5)	-77(3)	219 (3)	710 (5)	5 (1)
5)	55 (3)	256 (3)	514 (4)	4 (1)
7)	131 (3)	223 (3)	272 (4)	4 (1)
8)	180 (3)	148 (3)	-111 (5)	4 (1)
91)	247 (4)	305 (3)	- 74 (6)	7 (1)
92)	105 (4)	306 (3)	-64 (5)	7 (1)
93)	208 (4)	319 (3)	99 (6)	7 (1)
101)	375 (4)	154 (3)	32 (5)	7 (1)
102)	342 (4)	157 (3)	207 (6)	7 (1)
103)	323 (4)	57 (4)	103 (5)	7 (1)
111)	-247(3)	-100(3)	351 (4)	5 (1)
112)	-304(3)	-60(2)	513 (4)	4 (1)
121)	-464(4)	- 51 (4)	293 (6)	7 (1)
122)	- 440 (5)	50 (5)	377 (7)	10(1)
123)	- 389 (6)	18 (4)	174 (8)	11 (1)
	• • •			

Table 1. Parameters of non-hydrogen atoms and their estimated standard deviations

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	Anisotro	pic thermal f	actors are of t	he form: exp	$\sum_{i=1}^{3} \sum_{j=1}^{3} \sum_{i=1}^{3} \sum_{j=1}^{3} \sum_{i$	Bijhihjai*aj*].				
Positional parameter $\times 10^4$					Thermal parameter $\times 10^2$					
x/a	v/b	z/c	B_{11}	B ₂₂	B33	B ₁₂	B_{13}	B ₂₃		
0	0	0	255 (1)	249 (1)	314 (1)	-10(1)	53 (1)	-16(1)		
-1138(2)	72 (1)	1767 (2)	319 (7)	328 (8)	357 (7)	- 51 (7)	117 (6)	- 60 (7)		
950 (2)	1269 (2)	938 (3)	276 (8)	265 (9)	323 (9)	-43 (7)	48 (7)	-17 (7)		
-111(2)	1431 (2)	3466 (3)	320 (11)	283 (10)	333 (10)	26 (8)	56 (8)	4 (8)		
-1012(2)	643 (2)	3127 (3)	294 (10)	282 (10)	316 (10)	70 (8)	62 (8)	18 (8)		
-1853(3)	454 (2)	4338 (3)	351 (12)	319 (10)	378 (11)	57 (9)	106 (9)	31 (9)		
-1755(3)	1049 (2)	5771 (4)	471 (14)	437 (14)	380 (12)	35 (11)	188 (11)	1 (11)		
-874(3)	1830 (3)	6088 (4)	575 (16)	459 (14)	359 (12)	17 (13)	139 (11)	-95 (12)		
-64(3)	2012 (2)	4964 (4)	449 (13)	345 (11)	385 (11)	-17 (12)	72 (9)	- 56 (11)		
776 (3)	1689 (2)	2340 (3)	341 (11)	284 (10)	342 (11)	- 50 (9)	24 (8)	-42 (8)		
1967 (3)	1706 (2)	49 (4)	369 (12)	374 (12)	396 (12)	-130 (9)	129 (10)	-65(10)		
1919 (4)	2864 (3)	-87(6)	627 (21)	417 (16)	599 (19)	- 168 (15)	230 (17)	25 (15)		
3227 (4)	1325 (4)	897 (7)	378 (16)	708 (25)	813 (26)	- 24 (16)	201 (16)	87 (21)		
-2830(3)	- 393 (3)	4046 (4)	411 (14)	406 (13)	472 (14)	-18(11)	187 (11)	21 (11)		
- 4052 (4)	-41(4)	3014 (7)	473 (18)	648 (20)	821 (18)	-130 (14)	42 (15)	12 (18		
	Position x/a 0 - 1138 (2) 950 (2) - 1012 (2) - 1012 (2) - 1853 (3) - 1755 (3) - 874 (3) - 64 (3) 776 (3) 1919 (4) 3227 (4) - 2830 (3) - 4052 (4)	AnisotroPositional parameter x/a y/b 00-1138 (2)72 (1)950 (2)1269 (2)-111 (2)1431 (2)-1012 (2)643 (2)-1853 (3)454 (2)-1755 (3)1049 (2)-874 (3)1830 (3)-64 (3)2012 (2)776 (3)1689 (2)1967 (3)1706 (2)1919 (4)2864 (3)3227 (4)1325 (4)-2830 (3)-393 (3)-4052 (4)-41 (4)	Anisotropic thermal fPositional parameter $\times 10^4$ x/a y/b z/c 000-1138 (2)72 (1)1767 (2)950 (2)1269 (2)938 (3)-111 (2)1431 (2)3466 (3)-1012 (2)643 (2)3127 (3)-1853 (3)454 (2)4338 (3)-1755 (3)1049 (2)5771 (4)-874 (3)1830 (3)6088 (4)-64 (3)2012 (2)4964 (4)776 (3)1689 (2)2340 (3)1967 (3)1706 (2)49 (4)1919 (4)2864 (3)-87 (6)3227 (4)1325 (4)897 (7)-2830 (3)-393 (3)4046 (4)-4052 (4)-41 (4)3014 (7)	Anisotropic thermal factors are of tPositional parameter $\times 10^4$ x/a y/b z/c B_{11} 000255 (1) -1138 (2)72 (1)1767 (2)319 (7)950 (2)1269 (2)938 (3)276 (8) -111 (2)1431 (2)3466 (3)320 (11) -1012 (2)643 (2)3127 (3)294 (10) -1853 (3)454 (2)4338 (3)351 (12) -1755 (3)1049 (2)5771 (4)471 (14) -874 (3)1830 (3)6088 (4)575 (16) -64 (3)2012 (2)4964 (4)449 (13)776 (3)1689 (2)2340 (3)341 (11)1967 (3)1706 (2)49 (4)369 (12)1919 (4)2864 (3) -87 (6)627 (21)3227 (4)1325 (4)897 (7)378 (16) -2830 (3) -393 (3)4046 (4)411 (14) -4052 (4) -41 (4)3014 (7)473 (18)	Anisotropic thermal factors are of the form: expPositional parameter $\times 10^4$ x/a y/b z/c B_{11} B_{22} 000255 (1)249 (1)-1138 (2)72 (1)1767 (2)319 (7)328 (8)950 (2)1269 (2)938 (3)276 (8)265 (9)-111 (2)1431 (2)3466 (3)320 (11)283 (10)-1012 (2)643 (2)3127 (3)294 (10)282 (10)-1853 (3)454 (2)4338 (3)351 (12)319 (10)-1755 (3)1049 (2)5771 (4)471 (14)437 (14)-874 (3)1830 (3)6088 (4)575 (16)459 (14)-64 (3)2012 (2)4964 (4)449 (13)345 (11)776 (3)1689 (2)2340 (3)341 (11)284 (10)1967 (3)1706 (2)49 (4)369 (12)374 (12)1919 (4)2864 (3)-87 (6)627 (21)417 (16)3227 (4)1325 (4)897 (7)378 (16)708 (25)-2830 (3)-393 (3)4046 (4)411 (14)406 (13)-4052 (4)-41 (4)3014 (7)473 (18)648 (20)	Anisotropic thermal factors are of the form: $\exp \left[-\frac{1}{4} \sum_{i=1}^{3} \sum_{j=1}^{3} I_{i=1}^{3} I_{i=1}^{3} \sum_{j=1}^{3} I_{i=1}^{3} I_{i=1}^{3} \sum_{j=1}^{3} I_{i=1}^{3} I_$	Anisotropic thermal factors are of the form: $\exp \left[-\frac{1}{4} \sum_{i=1}^{3} \sum_{j=1}^{3} B_{ij}h_ih_ja_i^*a_i^*\right]$.Positional parameter × 104Thermal parameter × 102 x/a y/b z/c B_{11} B_{22} B_{33} B_{12} 000255 (1)249 (1)314 (1)-10 (1)-1138 (2)72 (1)1767 (2)319 (7)328 (8)357 (7)-51 (7)950 (2)1269 (2)938 (3)276 (8)265 (9)323 (9)-43 (7)-111 (2)1431 (2)3466 (3)320 (11)283 (10)333 (10)26 (8)-1012 (2)643 (2)3127 (3)294 (10)282 (10)316 (10)70 (8)-1853 (3)454 (2)4338 (3)351 (12)319 (10)378 (11)57 (9)-1755 (3)1049 (2)5771 (4)471 (14)437 (14)380 (12)35 (11)-874 (3)1830 (3)6088 (4)575 (16)459 (14)359 (12)17 (13)-64 (3)2012 (2)4964 (4)449 (13)345 (11)385 (11)-17 (12)776 (3)1689 (2)2340 (3)341 (11)284 (10)342 (11)-50 (9)1967 (3)1706 (2)49 (4)369 (12)374 (12)396 (12)-130 (9)1919 (4)2864 (3)-87 (6)627 (21)417 (16)599 (19)-168 (15)3227 (4)1325 (4)897 (7)378 (16)708 (25)813 (26)-24 (16)-2830 (3)-393 (3)4046 (4)411	Anisotropic thermal factors are of the form: $\exp \left[-\frac{1}{4} \sum_{i=1}^{3} \sum_{j=1}^{3} B_{ij}h_ih_ja_i^*a_i^*\right]$. Positional parameter × 104 x/a y/b z/c B_{11} B_{22} B_{33} B_{12} B_{13} 0 0 0 0 255 (1) 249 (1) 314 (1) -10 (1) 53 (1) -1138 (2) 72 (1) 1767 (2) 319 (7) 328 (8) 357 (7) -51 (7) 117 (6) 950 (2) 1269 (2) 938 (3) 276 (8) 265 (9) 323 (9) -43 (7) 48 (7) -111 (2) 1431 (2) 3466 (3) 320 (11) 283 (10) 333 (10) 26 (8) 56 (8) -1012 (2) 643 (2) 3127 (3) 294 (10) 282 (10) 316 (10) 70 (8) 62 (8) -1853 (3) 454 (2) 4338 (3) 351 (12) 319 (10) 378 (11) 57 (9) 106 (9) -1755 (3) 1049 (2) 5771 (4) 471 (14) 437 (14) 380 (12) 35 (11) 188 (11) -874 (3) 1830 (3) 6088 (4) 575 (16) 459 (14) 359 (12) 17 (13) 139 (11) -64 (3) 2012 (2) 4964 (4) 449 (13) 345 (11) 385 (11) -17 (12) 72 (9) 776 (3) 1689 (2) 2340 (3) 341 (11) 284 (10) 342 (11) -50 (9) 24 (8) 1967 (3) 1706 (2) 49 (4) 369 (12) 374 (12) 396 (12) -130 (9) 129 (10) 1919 (4) 2864 (3) -87 (6) 627 (21) 417 (16) 599 (19) -168 (15) 230 (17) 3227 (4) 1325 (4) 897 (7) 378 (16) 708 (25) 813 (26) -24 (16) 201 (16) -2830 (3) -393 (3) 4046 (4) 411 (14) 406 (13) 472 (14) -18 (11) 187 (11) -4052 (4) -41 (4) 3014 (7) 473 (18) 648 (20) 821 (18) -139 (14) 42 (15)		

least-squares refinement was taken from a plot of r.m.s. ΔF vs F_o to be $\sqrt{w} = 1/(0.70-0.0053 F_o)$ for unobserved reflections for which $F_c > F_o$ and for observed reflections and w=0 for unobserved reflections for which $F_c \leq F_o$. The final value of R = 0.023 was then reached in two cycles of anisotropic refinement of the non-hydrogen atoms and four cycles of isotropic refinement of the hydrogen atoms. All reflections with $\sin \theta/\lambda > 0.48$ were omitted during the refinement of the hydrogen atoms. In the final cycle of non-hydrogen

Table 3. Observed and calculated structure factors Columns are h, $10|F_o|$, and $10F_c$. Unobserved reflections are marked with*.

+,0,0 2 745 734 4 111 324 8 305 313 10 266 262 17 266 262	H,14,0 0 748 249 1 410 -11 2 285 289 3 420 5 4 185 188 5 400 00	-* 180 -9 37 10 298 -10 197 11 41 -11 40	177 300 293 186 -7 -7	H,0,7 1104 1135 534 547 179 -170 683 697 1203 1213 720 737 120 873	-> 2/9 284 -> 415 419 -6 96 99 -6 57 57 -7 178 170 -7 288 289 -8 355 -12	1 6 6 7 7 8 8 0 1 7 8 9 0 0 0 0 0 0 0 0 0 0 0 0 0 0 0 0 0 0	1111111	-10	221 221 260 263 41. 35 137 134 4.4. 135		47 32 450 441 213 501 280 -81 346 351 346 377	11111	50 -59 228 223 253 250 41- 12 39- 12 155 152 66 63		100 100 100 100 100 -103 107 211	-19	138 113 141 1-3 1,1,6 191 193	-10	172 162 H,1.7 344 -15 313 317 182 183 354 -16		152 147 38 -1 39 257 246 257 247 147
H+1+C L 159L 15A0 2 200 = 3 3 658 650 4 280 = 27 5 416 405 6 380 = 24	H,15,0 1 237 247 3 239 226 5 1 50	0 76 1 753 -1 755 -2 107 -2 107 -3 500	-73 -10 744 -10 732 -10 98 -1 419 375	216 218 233 243 255 265 318 327 142 127 220 223 H,1,2	-0 240 235 -0 200 3 -10 170 104 -11 170 104 -11 170 104	-10 1 -10 1 -11 10 -11 10 -11 10 -12 4	2113		10 14 300 300 13* -38 32* -14 300 304 300 304 100 30		40 201 201 356 301 376 - 13 199 204 116 322 416 420		270 275 38 11 90 86 187 193 213 210 39 15 53 45	-10	377 748 247 748 4.7.5		1000		261 264 37• 21 210 209 332 334 37• 36	·***	4124 H.2.8
+ 178 108 + 178 108 + 15 415 10 444 31 11 327 330 12 55 23 +,2,0	H.0.1 0 670 673 2 243 244 -2 549 555 -4 576 587 -6 310 323 -6 547 567		-113 -	3/5 - 3/3 404 409 1029 1043 76 71 494 199 638 646 665 670 297 287	-1 172 167 -1 172 167 -2 378 364 -3 78 364 -3 158 -155 -4 251 251 -4 450 451 -5 166 170	C 501 -1 11 -1 5 -2 89 -3 35 -3 35 -3 57	00300 1270 1270 1270 1270		40 -41 720 213 301 303 38 -10 157 150	-12	223 225 4214 4214 4214 4214	17797	130 141 10 141 10 - 6 390 - 39 132 127 410 - 0 H-13,4		215 217 314 317 314 20 215 20	-10		-10	109 299 300 299 105 159 41- 13 H,2.7		137 147 197 197 399 21 377 197 1225 225 409 107
0 1101 1083 1 155 - 158 2 553 556 3 56 30 4 333 3/7 5 //* - 22 6 3 0 369 7 66 51	-10 213 223 -10 338 336 -10 200 212 12 213 237 -12 109 134 H,1,1	-10 -11 -11 -11 -11 -11 -11 -11 -11 -11	-11 338 2AU -8 233 196 -	76 71 547 545 846 832 177 71 180 165 351 365 470 474 470 474				-10	4141 4.10.1	1	778 -98 91 418 -98 418 475 155 164	3	47 59 269 207 174 -1 100 101 145 145 180 172		303 308 38- 14 3628 188 18- 252 251 62 56 83 87 260 256		H.4.6 451 458 61 59 33. 26 333 339 307 315		67 33 72 823 315 282 51 50 346 -23 197 195 199 313		232 224 40 222 163 158 H, 3, 8 55 43 151 143
10 320 317 11 424 34 12 160 152 +.3+0 1 148 147	0 392 -355 1 1030 1024 -1 425 425 2 766 -767 -2 360 350 3 1013 1030 -3 567 531 4 151 150	0 694	-704 -0 -704 -0 530 -1 530 -1 529 -1 45 -1	300 105 179 192 38 - 31 208 202 281 290 41 - 8	10 142 137 -10 187 187 -11 50 56 -11 50 56 -11 50 56 -11 50 56			201010	202 201 12 -72 100 -44 267 275 235 235 61 71 203 196		402 407 1814 1814 729 228 136 346 229 228 136 346 19 191	-3 -[103 103 2.14.4 174 171 40. 171 79 78 124 119	-10	4,8,5 241 243 46 -71 274 257 375 383	244429977	104 100 106 138 104 103 144 103 268 267 70 91		35. 16 148 151 287 277 3835 243 250 60 133		177 149 177 149 177 149 177 149
2 520 510 3 334 329 4 449 427 5 336 531 6 145 147 7 574 570 1 251 250			-14 -14 -244 -25 -25	*.2.2 254 250 274 17 270 17 270 17	-1 1/C 166 -2 50 61 -2 58 54 -3 50 51 -4 1/8 451 -4 1/8 1/6	H, 3 C 12 -1 16 -2 19 -3 53 -3 53			122 129 144 129 144 147 40+ -31	-1			170 165 41. 19 ***C** *31 535 82 101 332 333		109 109 268 277 273 275 360 21 274 226 276 276	-10 -10	142 139 234 238 211 708 H.5.6 79 91		H.3.7 45 -36 325 325 355 364 81 88 51 -49		179 174 400 -8 H,4,6 169 160
11 163 161 17 45 43 H.4.C 0 667 654 1 165 160 2 688 683	-10 63 -51 -11 250 251 -11 750 251 -11 76 -29	-# 281 -9 50 -10 250 -10 232 -11 425		730 652 700 759 315 290 419 445 515 541	A 90 91 -A 35. 10 -7 245 251 -7 255 256 -8 381 -9 204 194 -9 229 221		5210 5223		1/2 0 - 1/2 1/2 0 - 1/2 0 - 1/2 1/2 0 - 1/2 0 - 1/2 1/2 0 - 1/2 0		31 -22 501 500 501 500 428 430 309 -29 428 430 399 397 113 115		533 533 375 334 379 336 173 171 145 150 119 116 264 263 108 117		107. 105 107. 105 107. 105		399 405 3426 33. 214 310 309 362 25 51 219 218		356 358 37* -28 56 61 151 143 303 301 37* -30 164 152	4144444	175 167 196 190 378 17 160 145 280 278 260 278
4 497 505 5 139 -128 6 368 364 7 405 19 4 433 433 1 75 -85 10 255 255 10 255 -35	H,2.1 0 416 415 1 942 -927 -1 214 213 2 1679 1659 -2 1155 1153 3 456 -440	0 12/ 1 469 -1 481 -2 91 -3 55 -1 35P	-123 476 -91 234 -11 234 -11	228 210 345 355 165 16 355 4 123 121 123 121 52 49	H,1C,2 0 346 344 1 344 26 -1 44 -47 2 543 541 -2 247 293	19700112	12, 20, 2		240 275 247 244 70 86 39 36 113 111 141 116 410 -17	-78-499-100	233577		**,1.5 28* -15 383 397 577 587 30* 32 28* 2 176 183		104 104 242 244 244 230 144 147		30, 20 30, 80 107 104 791 299 410 - 26 208 206 43 37		239 267 169 166 H.4.7 329 328 60 -53		10 - 73 N+5.8 20 - 50 21 274 223 221 37 - 19
12 127 108 H1510 1 794 783 2 387 352 3 752 749 4 284 -12 5 475 475	-1 70 AR -1 723 713 -4 726 447 -5 717 712 -5 717 712 -6 632 617 -7 50 -15 -7 10 127		193 -12 193 -12	167 160 222 216 H.3.2 200 -204 651 657 238 738 273 271	-1 101 102 - 101 102 - 101 102 - 101 102 - 100 60 - 5 100 100 - 0 257 207 - 0 302 304 - 7 394 30	H,4, 0 38 1 110 -1 80 -2 20	401		261 266 10	-1,	4,6,4 4,6,4 4,1 4,53 92 83 98 83 98 83 224 530		465 463 113 111 59 321 315 321 379 383 15. 7 163 164 280 283		40. 24 370 172 159 271 259 271 259 189 181		H, 6.6 338 343 45 52 40. 32 258 255 263 265 3829		252 259 277 282 37• 2 158 150 301 305 38• 26 91 96	11111	177 164 225 221 48 39 206 212 138 136
6 32° 19 7 450 452 8 169 417 9 305 306 10 44° 48 11 196 194 12 45° 29		-0 220 -0 257 -10 41 -10 41 -10 41	210 -10 -10 -10	171 173 777 761 523 511 228 -220 148 127 186 192 568 569 112 118	-7 47 -60 -8 198 199 -8 248 244 9 4119 -7 4019 -7 4019		1,000 CO	111101	302 307 202 28 38 17 18 17 18 225 40 - 16		110 -11# 842 -331 -256 -11 120 -255 120 -255		10. 10 140 140 197 195 10. 3 236 245	0	2127 225 2127 225 2127 225 2127 225 2127 225 2127 225 2127 225 2127 225 2127 225 2125 215 215 215 215 215 215 215 215 215 215 215 215	1.4.1.60.	59 57 245 243 275 277 394 17 265 273 414 17		195 198 43 -56 199 198 40• 72 H.5.7	0	233 237 400 - 45 380 - 16 169 160 170 165 170 165
1135 1111 1 200 205 2 776 759 3 248 235 4 407 400 5 44 407	-12 133 184 H.3.1 0 247 -287 1 1225 1812 -1 500 482 -3 328 -154	-1 75	-15 291 - 338 - 51 - 320 - 295 - 10 295 - 10	290 286 406 411 57 53 745 236 175 173 179 - 28	-1 167 174 -1 167 174 -2 366 -17 -2 346 -1 -3 321 125 -3 232 242 -4 87 77	A 170 -8 25 -9 30 -10 16 -10 24 -11 35	1002 - 100		84 94 271 224 261 266 11. 48 272 276 201 197	-7	11 -62 15 212 160 162 160 -62 178 -18 228 221 42 -10		463 459 85 80 50 -53 361 368 485 491 90 96 30 -38	1007.8	400 -12 440 14 155 153 188 182 198 12 168 155	0	232 232 +.7.6 287 287 287 287 287 287		193 (94 35 - 20 35 - 20 193 195 38 - 107 193 197	100	111 175 185 183 H,7,8 405 192 152 150
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2 129 105 1 496 483 5 419 419 5 419 419 6 51 -114 8 315 316 9 310 309 10 69 72	-7 715 728 -7 715 728 -8 77 -71 -9 220 226 -9 506 556 -10 59 -67 -11 229 224	-10 169 -1, 11, 0 83 1 334 -1 351 -1 351 -2 56	101 170 140 334 334 	841 830 487 438 246 -272 87 88 484 984 511 523 145 -148 370 376	H+12,2 U 125 13C U 125 13C		200755555555555555555555555555555555555		100 15 113 216 216 216 400 11 229 225 40 41	******		-10	127 310 40+ 6 38 55 240 248 240 248 240 248 240 28		3919 134 127 136 126 136 126 118 116 1,12,5 248 251		53 61 167 162 240 240 171 170 M.B.C		189 183 44 -38 201 200 147 18. 18 36. 21 214 212	111-0	111 112 43 - 10 130 121 143 132
11 107 100 H+8+0 0 485 483 1 222 -207 2 524 520 3 58 58 4 302 30	-11 219 218 -12 410 -10 H.e.1 0 524 517 -1 76 -03	-1 2/4 -1 2/4 -5 28/ -5 3/6 -5 3/6 -5 3/8	224 17 14 276 384 -21 -16 -10 -10 -10 -10 -10 -10 -10 -10 -10 -10	638 631 338 -9 248 -240 182 182 182 182 182 182 183 30 164 29 259 257 147 149			248 13 120 224 14 24 185	-î		-10	149 -119 146 147 137 111 408	11111	5135 546 435 441 108 106 294 204 513 517 66 -60 107 98		19. 124 19. 156 19. 156 71 156 71 157 165 157		37 19 331 - 332 160 - 156 133 - 65 239 - 237 193 - 196		62 66 83 95 165 133 223 218 39* -2 184 183 H,7.7	0777408	201 144 127 145 147 141 200 187 151 153
4 324 322 7 364 -28 8 336 334 9 36 85 10 205 200 11 464 13	2 857 831 -2 568 563 3 231 213 -3 110 137 -4 681 646 -4 571 559 -5 48 -54	-7 299 -8 40 -8 40 -1 163 -1 163 -1 163	298 -12 163 1 1 1 1 1 1 1 1 1	414 31 394 11 174 166 P+5+2 175 167 681 982	-H 210 204 H+13+2 0 74 -71 1 199 203 -1 139 213 -1 139 -21	0 450 -1 51 -2 401	3 	14400000	628 623 672 690 667 690 667 690 667 690 667 690 667 690 669 160		153 157 153 157 155 -155 155 -155 155 -155 155 -155 155 -155 155 -155 155 -155		247 254 41 47 68 -66 132 336 310 313 19 48 187 180		155 151 11155 154 153 224 241		370 -16 137 134 196 192 390 30 262 257		420 -55 201 195 156 150 39 20 206 205 176 171		3912 145 161 214 204 3912 14 16 14 16 16 16C
1 369 382 2 35* 21 3 437 436 4 149 -149 5 316 314 6 48 63 7 362 367	-6 667 652 -7 64 52 -7 65 52 -7 7 65 52 -7 7 65 52 -7 7 65 52 -7 7 7 65 52 -7 7 7 7 7 7 7 7 7 7 7 7 7 7 7 7 7 7 7	-1 55 -2 258 -2 258 -3 36 -3 36 -3 30 -3 30 -4 30 27 -5 38	2027	404 - 78 25 - 16 900 - 974 159 - 152 57 - 52 516 - 575	-1 243 250 -1 243 250 -4 31 62 -5 193 186 -5 226 221 -6 416 -17		198 514 551 -188 -2592 541 -17	-i;	(+1+4 187 -17) 242 -313 606 -618 210 -207	100-71871	21110-1210-1210-1210-1210-1210-1210-121		526 536 337 10 217 214 518 514 518 514 74 78	111 - P	185 191 410 -2 1.0.6 369 176 375 153		40/ 264 267 182 18 31 -60 216 274 213 203		390 9 192 182 253 253 390 38 196 192 410 9	=: -!	3910 215 20F H.C.9 155 167 1911
10 41 21 H.10.7	-10 731 277 -11 399 -31 -12 100 100 -12 100 101 H.5.1	-5 18 6 222 -6 301 -7 400 -7 207 -8 207 -8 207 -8 207 -8 207 -8 207 -7 400 -8 207 -8 207 -7 400 -7 207 -7 400 -7 400	226 -6 3049 -7 21 -8 154 -8 207 -9 154 -8	32* 17 161 - 157 270 267 552 552 48 - 128 128 - 124 266 264 317 321 40 40	-/ 1/4 1/P H,14.7 0 167 164 1 194 2 -1 194 2 2 207 201		188 2263 105 150 257	1.1.1.1.1	1465 4 910 - 149 4 92 4 910 - 210 4 82 4 910 - 210 4 82 4 910 - 210	-1	200 - 10 200 - 201 200 - 10	11111	101 357 149 -49 114 355 149 -49 140 355 149 -49 140 355 149 -49 140 355 149 -49 149 -49 -49 149 -49 149 -49 14		325 362 396 401 269 269 398 436 258 256 337 367 208 212 79 32		132 133 166 162 42 40 238 232 42* -19 H-10+6		108 103 398 52 40 199 192 237 233 3825 10. 162	2011)(1)(1 39+ 34 190 183 148 133 H+3,9
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1 354 362 2 39 19 1 232 232 4 36 - 29 5 295 297 6 55 46 7 175 173	-5 638 626 -6 639 -10 -7 307 312 -7 444 439 -7 444			71 -61 629 617 239 -122 131 -122 175 -166 618 620 480 32 - 7	0 40 34 1 179 182 -1 201 198 2 41 17 -3 266 260 H.0.3			-12 , -]	244 264 200 -190 123 -123 147 189	10	109 - 119 109 - 10 108 - 10 108 - 10 108 - 10 109 - 110	-1,	++++		100 100 200 200 200 200 200 200		39. 11 187 173 41. 1 11.6 19. 14 19. 14 19. 19 19. 19		163 164 390 6 167 163 220 221 390 -11 163 162 410 -33	1111-0	H,4,9 102 83 97 86 406 -5 16C 158 408 -26 171 165
5 172 165 H,12,0 U 254 254 4 325 326	-10 34 -1 -10 34 -1 -11 324 117 -11 160 157 -12 160 157	0 258 1 39 -1 38 -2 215 -2 86 -3 60	263 -6 -31 -7 -31 -7 221 -8 -87 -9	310 -70 321 320 350 -17 167 -167 204 202 345 390 380 14	0 843 896 2 429 441 -2 595 600 4 508 526 -4 776 762 6 417 427 -6 556 371 -6 116 136	-9 191 -9 191 -10 -0 -11 19	1.1.5 A F F		781 700 77 - 30 329 339 349 400 31 40 143 141 313 329		41 -92 261 276 63 -61 211 211 417 452 211 -290		210 244 387 393 170 -167 46 45 108 102 84 95 17* -11 227 -227	-10 -11	4111 37. 10 272 288 1922 128 121 4.2.0		402 39. 36 LC7 99 174 167 187 182 P.12,6		H.10,7 177 165 55 51 194 +10 171 165 173 169 173 169		+,5,9 +7- 33 150 134 4019 163 155 401
4 185 196 5 410 186 7 140 138 7 420 4 H 187 177 H+13+0 1 253 25-	0 833 81 8 1 255 245 -1 268 -262 2 306 314 -2 466 464 -3 187 1/7 -3 265 250 4 256 250 -4 256 250			214 212 231 232 432 24 H.7.2	-R 14A 156 10 204 212 -10 383 397 -12 196 195 H.1.3 0 111 112		100000		111 111 111 111 111 111 111 111		245 245 109 105 109 105	-768090	151 154 175 184 185 184 202 197		107 - 27 411 - 27 134 - 114 132 - 204		213 204 41 -15 36 129 125 43 35 H,0,7	0,744	H.Q.8 203 208 241 237 241 237 241 237	×	H+0+10 126 121 94 80
2 410 1 3 246 249 4 380 1 7 200 194 6 30 194 7 178 171	-5 41 -51 -5 300 -25 -6 390 390 -6 468 467 -7 144 15 -7 144 15	-1 108 -1 150 -1 150 -1 150 -1 150 -1 150 -1 150 -1 150 -1 150	196 -1 196 -1 196 -1 196 -1 196 -1 196 -1 196 -1 197 -1 197 -1	435 445 294 424 40 440 328 328 351 358 85 86 154 -149	-1 625 635 -2 23. 19 -1 1018 1010 -1 1018 1010 -1 1018 1010 -1 1018 1010			-i -i	224 233 4.4.4 208 -196 187 199 614 615	-1,	1223-1223		**************************************		105 - 105 105 - 105 105 - 105 105 - 105 105 - 105 105 - 105	0~~~~~	301 304 267 276 96 -93 240 246 130 152 163 152 218 216	-10	151 256 151 117 H-1.8 113 109		

atom refinement the average shift/error for positional and thermal parameters was 0.03, with a maximum of 0.21. In the final cycle of hydrogen atom refinement the average shift/error was 0.03 with a maximum of 0.43.

The final parameters and their estimated standard deviations are listed in Tables 1 and 2. The σ 's were obtained by simultaneous refinement of all parameters except those of C(12), H(121), H(122), and H(123), using full data for non-hydrogen σ 's and limited data for hydrogen σ 's. For the four omitted atoms, which exceeded the capacity of the full-matrix least-squares program, σ 's were obtained by application of an empirical correction factor. The list of observed and calculated structure factors is given in Table 3. Examination of these structure factors by the method of Housty & Clastre (1957) did not show any detectable secondary extinction.

Discussion

The crystal consists of discrete molecules. Fig. l shows the projection of atomic coordinates on (010). Since the palladium atom is at a center of symmetry, it has strictly planar coordination.

Bond lengths and angles involving the heavy atoms are shown in Fig.2 and the C-H distances are listed in Table 4. The Pd-N bond length of 2.031 Å agrees well with the values of 2.030 and 2.043 Å in bis(ethylenediamine)palladium(II) chloride (Wiesner & Lingafelter, 1966), 2.022 Å in bis-(2,2'-dipyridyliminato)palladium (Freeman & Snow, 1965), and 2.01 Å in bis-(*N*n-butylsalicylaldiminato)palladium (Frasson, Panattoni & Sacconi, 1964). The Pd-O bond length of 1.991Å agrees well with the values of 2.00 Å in palladium(II) oxide (Pauling & Huggins, 1934) and 2.00 Å in bis-(*N*n-butylsalicylaldiminato)palladium (Frasson, Panattoni & Sacconi, 1964).

Table 4. Carbon-hydrogen bond distances and their e.s.d.'s

C(4) - H(4)	0·92 (4) Å	C(10) - H(101)	0·83 (5) Å
C(5) - H(5)	0.93 (4)	C(10) - H(102)	0.98 (5)
C(6) - H(6)	0.97 (4)	C(10) - H(103)	0.99 (5)
C(7) - H(7)	0.92 (4)	C(11) - H(111)	1.00 (4)
C(8) - H(8)	0.96 (4)	C(11) - H(112)	0.97 (4)
C(9)-H(91)	0.88 (5)	C(12) - H(121)	0.87 (5)
C(9) - H(92)	1.00 (4)	C(12) - H(122)	1.03 (6)
C(9)-H(93)	0.96 (5)	C(12)-H(123)	1.09 (6)

The C-C bonds of the salicylaldimine residue again have the trend which has been found for the average bond lengths of eight salicylaldimine chelates (Lingafelter & Braun, 1966); namely, the interatomic distances C(3)-C(4), C(4)-C(5), and C(5)-C(6) are less than 1.386 Å, while the interatomic distances C(1)-C(2), C(2)-C(3), C(6)-C(1), and C(1)-C(7) are greater than 1.409 Å. The small average standard deviation of 0.005 Å for a C-C bond in this heavy metal chelate illustrates the good precision which can be achieved with diffractometer data, even with the use of Mo K α radiation without balanced filters.



Fig. 2. Interatomic distances and angles. Average values of the estimated standard deviations are: Pd-O, Pd-N, 0.002 Å; O-C, N-C, 0.003 Å; C-C, 0.004 Å; O-Pd-N, 0.1°; Pd-O-C, 0.2°; O-C-C, N-C-C, C-C-C, 0.3°.

Least-squares equations for selected planes within the molecule and the angles between these planes are listed in Tables 5 and 6. The effective 'step' of 0.42 Å between the parallel salicylaldimine planes of the molecule is appreciably less than the step of 0.76 Å in the related planar nickel chelate, 3-M-Ni. In addition, while atoms N and C(7) in the latter nickel chelate were found to have large deviations (0.187 and 0.170 Å, respectively) from the plane of the benzene ring, in this case the deviations of these two atoms are significantly less (0.024 and 0.007 Å respectively). Thus the entire salicylaldimine group is quite closely planar.

Table 5.	Coefficients of least-squares plane equations
	$A(x/a) + B(y/b) + C(z/c) = D^*$

11(34	μ	$\mathcal{O}(\mathcal{L}/\mathcal{C})$	P	
Plane	A	В	С	D *
Coordination	- 5.939	7.497	-4.130	0
Benzene	-6.317	8.493	- 3.106	0.210
Salicylaldimine	-6.286	8.529	- 3·107	0.213
Isopropyl	-4.875	0.684	7.546	- 0.806
C(3) - C(11) - C(12)	- 5.089	3.397	7.186	4·214
* 5	• • . •		18	

D = origin-to-plane distance (Å).

Table 6. Angles between least-squares planes

Plane (1)	Plane (2)	Angle
Coordination	Benzene	8.6°
Coordination	Salicylaldimine	8.7
Salicylaldimine	Benzene	0.5
Salicylaldimine	Isopropyl	83.1
Benzene	Isopropyl	83·2
Benzene	C(3) - C(11) - C(12)	87.1

The non-bonded intramolecular steric interactions which cause a species of tetrahedral configuration to appear in equilibrium (Holm & Swaminathan, 1963) with a species of planar configuration for solutions of bis-(*N*-isopropyl-3-alkylsalicylaldiminato)nickel chelates have been described in detail from the crystal structure of 3–M–Ni (Braun & Lingafelter, 1966). Further confirmation of these steric interactions is given by the non-bonded intramolecular distances listed in Table 7. That is, the unfavorable steric interactions involving the hydrogen atoms of the isopropyl group are again evident, particularly in the distances of 2·19 Å for H(8)---O', compared with the sum of the van der Waals radii of 2·6 Å (Pauling, 1960), and the distance of 2·11 Å for H(7)---H(93), compared with the radii sum of 2·4 Å. In the planar chelate 3–M–Ni these distances were 2·20 and 2·2 Å, respectively, while in the tetrahedral chelate 3–E–Ni they were > 4·51 and > 2·9 Å, respectively.

The most important result of this structure determination is the characterization of the steric interactions between the 3-alkyl substituent and the Nisopropyl group. This is important in examining the apparent increase in the stability of the tetrahedral species of the nickel chelates in toluene solution (Holm & Swaminathan, 1963) as the 3-substituent is changed from hydrogen to methyl to ethyl. Although Holm & Swaminathan (1963) reported that metal chelate scale models indicate unfavorable interaction in a planar structure between the isopropyl group and the 3-substituent, only slight steric interaction between these groups is found in the crystal structures. Table 8 summarizes the non-bonded intramolecular contacts between these two groups for the three 3-alkyl chelates whose structures are known: 3-M-Ni, 3-E-Ni, and 3-E-Pd. The two independent distances in 3-E-Ni are listed separately because of the marked difference in the positions of the two 3-ethyl groups.

Examining first the contacts involving atom C(11), it is seen that the C(11)---C(9') distances are normal in the tetrahedral and planar chelates. Although the C(11)---C(10') distances of 3.71 Å in 3-M-Ni and 4.10 Å in 3-E-Pd are quite close to the radii sum of

	The primed atoms are related to the unprimed atoms by the center of symmetry.							
	C(9)	H(91)	H(92)	H(93)	C(10)	H(101)	H(102)	H(103)
C(7)	2.878	3.71	3.02	2.71	3.042	3.77	2.86	3.29
H(7)	2.56	3.4	2.9	2.1	2.92	3.6	2.5	3.4
H(8)	1.98	2.2	2.2	2.8	2.06	2.2	2.9	2.4
O′	4.110	4.36	4.19	4.84	3.391	3.70	4.22	3.04
C(11')	4.717	4.42	4.96	*	4.101	3.80	*	4.03
H(111')	3.77	3.5	4 ·0	4.7	3.53	3.2	4.5	3.6
H(112')	*	4.9	*	*	4.89	4.5	*	4.9
C(12')	*	4.74	*	*	3.762	3.35	4.66	3.54
H(121')	4.99	4.5	*	*	3.76	3.2	4.6	3.7
H(122')	*	*	*	*	4.75	4.3	*	4.4
H(123')	4.77	4.6	*	*	3.04	2.8	3.9	2.6
	C (11)	H(111)	H(112)	C(12)	H(121)	H(122)	H(123)	
0	2.807	2.55	3.70	3.400	4.05	4.07	2.94	
C(8')	3.920	3.11	4.68	4.149	4.27	*	3.62	
H(8')	3.08	2.2	3.8	3.56	3.8	4.5	3.2	
H(4)	2.62	3.5	2.3	3.42	4.0	3.0	4.1	
	C(8')	H(8′)	N					
0	2.815	2.19	2.875					

Table 7. Intramolecular distances (Å)

* Distance is greater than 5.0 Å.

	Tetral	hedral		
	3-E-Ni	3-E-Ni	Pla	nar
C (11)	I	11	3-M-Ni	3-E-Pc
C(11)C(8')	*	*	3 ·77	3.92
C(11)C(9')	4.66	4.82	4·79	4·72
C(11) - C(10')	*	*	3.71	4.10
Minimum between				
H[C(11)] H(8')	4.74	*	2.3	2.2
Minimum between				
H[C(11)] H[C(9')]	3.1	3.8	3.5	3.5
Minimum between				
H[C(11)]H[C(10')]	4.4	4.6	2.8	3.2
C(12)				
C(12) - C(8')	*	*		4.15
C(12) - C(9')	*	4.25		*
C(12) - C(10')	*	*		3.76
Minimum between				
H[C(12)]H(8')	*	*		3.2
Minimum between				
H[C(12)] H[C(9')]	*	2.6		4.5
Minimum between				
H[C(12)]H[C(10')]	*	3.8		2.6
* 0	reater than	5·0 Å.		

 Table 8. Intramolecular contacts between the N-isopropyl group and the 3-alkyl substituent (Å)

4.0 Å, neither of these contacts is seriously close, since the H---H distances between C(11) and C(10') are >2.8 Å for 3-M-Ni and >3.2 Å for 3-E-Pd, well above the radii sum of 2.4 Å. The only evidence of unfavorable steric interaction in the planar chelates involving atom C(11) is found in the C(11)---C(8') contact. That is, the C(11)---C(8') distances of 3.77 Å in 3-M-Ni and 3.92 Å in 3-E-Pd are slightly less than the radii sum of 4.0 Å. This intramolecular contact may indeed be of some steric importance, since the H(111) ---H(8') distances of 2.3 and 2.2 Å in these two planar chelates are also slightly less than the radii sum of 2.4 Å.

Examining next the contacts involving atom C(12), it is seen that the C(12)---C(8') distances and the C(12)---C(9') distances are normal in both 3-E-Ni and 3-E-Pd, but that the C(12)---C(10') distance in 3-E-Pd is 3.76 Å, slightly less than the radii sum of 4.0 Å. This is probably not a seriously close contact, since the minimum H---H distance between the C(12) methyl group and the C(10') methyl group is 2.6 Å between H(123) and H(103'), well above the radii sum of 2.4 Å. In view of the absence of any seriously close intramolecular contacts involving the 3-substituent, it is likely that the steric interactions indicated for a planar structure by the scale models are in actuality somewhat alleviated by the sizable step between the parallel salicylaldimine residues.

Even though the intramolecular contacts involving the 3-substituent are not seriously close, they are close enough in the planar chelates to severely restrict free rotation of the methyl and ethyl groups. Among these chelates, the entropy increase which would be achieved by the increased rotational freedom of the 3-substituent when the coordination configuration is changed from planar to tetrahedral would likely follow the sequence hydrogen < methyl < ethyl. Thus, the increasing stability of the tetrahedral species in solution as the 3-substituent is changed from hydrogen to methyl to ethyl would be explained by an entropy effect.

Examination of the intermolecular contact distances shows only one which requires comment: the distance of 3.619 Å between C(12) and C(10) is less than the sum of the radii for two methyl groups, 4.0 Å. This apparently close intermolecular contact is explicable in terms of the high thermal parameters of C(10) and C(12). The remaining distances are normal, the closest intermolecular contacts being 2.5 Å for H---H, 2.98 Å for O---H, 3.56 Å for N---H, 2.90 Å for C(sp^2)---H, 3.13 Å for C(sp^3)---H, 3.536 Å for C(sp^2)---C(sp^2), 3.735 Å for C(sp^2)---C(sp^3).

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