at *ca.* 225–228°, the same decomposition temperature observed for the optically inactive compound.

The rotations of all the optically active derivatives described above at 546 and 578 mμ are presented in Table X. The ORD curves of these compounds are shown in Figure 14.

Degradation of $(Me_2)_2Ni$-*C* Derivatives. A tetrahydrofuran solution containing 0.145 g (0.38 mmol) of $(Me_2)_2Ni^{IV}$-*C* with an excess of sodium amalgam was allowed to stir under nitrogen for 2 days. The resulting black mixture was poured into water, the tetrahydrofuran removed *in vacuo*, and the aqueous solution treated with $(CH_3)_4NCl$. Fractional recrystallization of the crude precipitate from acetone–water mixtures afforded 0.0346 g (0.147 mmol, 20%) of $(CH_3)_4N[(3)\text{-}1,2\text{-}B_9H_{10}C_2(CH_3)_2]$ and a very small amount (0.006 g, 0.013 mmol) of red $(CH_3)_4N[(Me_2)_2Ni^{III}\text{-}A]$, which was probably formed *after* the degradation of the series C derivative.

In another reaction, 0.249 g (0.594 mmol) of $K[(Me_2)_2Ni^{III}\text{-}C]$, 2 ml of hydrazine hydrate, and 1 g of sodium cyanide in an ethylene glycol–water solution were heated in a steam bath. Solid NaOH pellets (0.7 g) were slowly added and the heating continued for 1 hr. The mixture was diluted, treated with $(CH_3)_4NCl$, and the crude precipitate fractionally crystallized from ethanol–water to afford 0.060 g (0.255 mmol, 21.4%) of $(CH_3)_4N[(3)\text{-}1,2\text{-}B_9H_{10}C_2(CH_3)_2]$. In this experiment and in the one described above, the degradation products were identified by their proton nmr spectra and/or infrared spectra.

Thermal Rearrangement of Unsubstituted $(H_2)_2Ni^{IV}$. In a high vacuum system, $(H_2)_2Ni^{IV}$ was allowed to slowly sublime through a 9-in. glass tube which was heated to 360–400°. An oil bath at the bottom of the assembly was maintained at 150°. The level of the oil bath was always just below the level of the $(H_2)_2Ni^{IV}$ sublimate so as not to force too much through the hot column at once; otherwise, serious decomposition occurred. The process was allowed to continue for 1 week, after which time a small amount (*ca.* 0.2 g) of yellow material had condensed at the top of the strongly heated portion of the tube. This material was crystallized from dichloromethane–hexane to afford a small amount of crystalline yellow-orange flakes as the least soluble fraction. It was this fraction that contained the majority of the rearranged products, the more soluble fractions containing considerable amounts of starting material. The cyclic voltammogram of the least soluble material is shown in Figure 18, and the potential data are presented in Table VII.

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The Crystal Structure of 3,3′-*commo*-Bis[undecahydro-1,2-dicarba-3-nickela-*closo*-dodecaborane], a Nickel(IV) Complex of the Dicarbollide Ion[1]

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Abstract: The crystal structure of the neutral molecule, 3,3′-*commo*-bis[undecahydro-1,2-dicarba-3-nickela-*closo*-dodecaborane], $Ni(B_9C_2H_{11})_2$, has been determined from 3242 independent X-ray data obtained from a single crystal using a scintillation counter and the θ–2θ scanning technique. The monoclinic unit cell has dimensions $a = 13.371$, $b = 10.398$, $c = 13.556$ Å, and $\beta = 119.16°$. The space group is $P2_1/c$. There are four molecules in the unit cell and the calculated density is 1.31 g/cc. The model was refined to a conventional discrepancy factor of $R = 0.035$. The molecule has the shape of two icosahedra, each composed of nine boron atoms, two carbon atoms, and a nickel atom, which have one vertex position occupied by the nickel shared in common. The positions of the carbon atoms in the cage were determined and all hydrogen atoms were located. The carbon atoms in opposite cages are as close to each other as is possible in the staggered configuration. The molecule has point symmetry C_2. There are two enantiomorphic rotational isomers present in the crystal.

Another in the series of transition metal complexes of the dicarbollide ion, $(B_9C_2H_{11})^{2-}$, has been prepared.[2,3] This complex, $Ni(B_9C_2H_{11})_2$, 3,3′-*commo*-bis[undecahydro-1,2-dicarba-3-nickela-*closo*-dodecaborane],[4] is a neutral molecule in which the nickel is in a formal 4+ valence state. Warren and Hawthorne predicted the structure as having the nickel ion sandwiched between the open fivefold faces of two $(B_9C_2H_{11})^{-2}$ icosahedral fragments. Because of the unusual chemical and spectral properties of this compound, such as the high dipole moment and two low-field doublets in the 32.0-Mc/sec ^{11}B nmr spectrum, observed for this molecule and an analogous $Pd(B_9C_2H_{11})_2$ neutral molecule,[5] an unusual structure was expected.

In this paper are reported the results of a single-crystal X-ray analysis which confirms the sandwich type structure in the molecule, shown in Figure 1. We show that there are two enantiomorphic rotational configurations present in the crystal and that the cages are staggered. The structure is novel among the unsubstituted metallocarboranes containing two dicarbollide ions, in that the pairs of carbon atoms in opposite cages are as

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[1] Work done under the auspices of the U. S. Atomic Energy Commission.

[2] L. F. Warren and M. F. Hawthorne, *J. Amer. Chem. Soc.*, **89**, 470 (1967).

[3] M. F. Hawthorne, D. C. Young, T. D. Andrews, D. V. Howe, R. L. Pilling, A. D. Pitts, M. Reintjes, L. F. Warren, Jr., and P. A. Wegner, *ibid.*, **90**, 879 (1968).

[4] This name is based on the nomenclature rules given in *Inorg. Chem.*, 7, 1945 (1968). The name given by Warren and Hawthorne in their original publication, ref 2, is bis-π-(3)-1,2-dicarbollylnickel(IV).

[5] L. F. Warren and M. F. Hawthorne, *J. Amer. Chem. Soc.*, **90**, 4823 (1968).

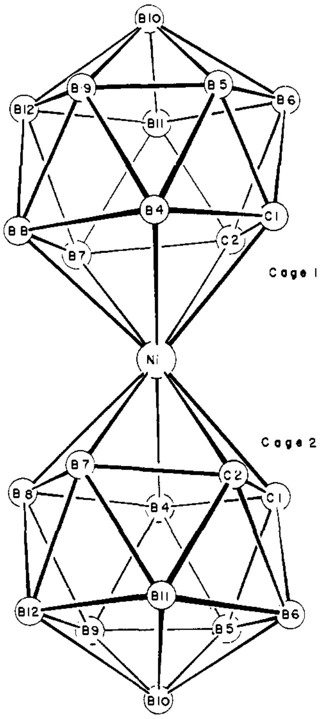

Figure 1. Skeletal drawing of $Ni(B_9C_2H_{11})_2$ showing the numbering system used (hydrogen atoms not shown).

close to each other as is possible in this staggered configuration, so that the molecule has only the symmetry of a twofold axis. A similar configuration of carbon atoms has been observed, however, in a double icosahedral-cage cobalt metallocarborane, in which the two cages are linked by a S–C–S bridge,[6] and in a metallocarborane containing three linked icosahedra.[7]

Experimental Section

The yellow crystals, as sent to the authors by Professor Hawthorne of the University of California, Riverside, were used. One of dimensions 0.17 × 0.15 × 0.12 mm was glued to the tip of a glass fiber in the open air with its *b* axis parallel to the rotation axis of the fiber.

Approximate cell dimensions and space group possibilities were determined from oscillation and Weissenberg photographs taken with copper radiation (Cu $K\alpha_1$; λ 1.5405 Å). Space group absences were checked and from measurements on the *h*00, 0*k*0, and 00*l* reflections, accurate unit cell dimensions were determined using a manually operated General Electric XRD-5 diffractometer. Intensity data were collected using a card input, card output automated General Electric diffractometer equipped with a quarter circle, Eulerian cradle goniostat. Cu $K\alpha$ radiation from an X-ray tube operated at 35 kV and 16 mA was used. A 0.003-in. thick nickel $K\beta$ filter was placed between the crystal and the receiving slit. The intensity of the scattered radiation was measured with a scintillation counter equipped with a pulse height discriminator. The integrated intensity of each reflection was measured by the θ–2θ scan technique, by scanning at a rate of 1°/min from 0.65° below the 2θ angle at which $K\alpha_1$ was diffracted, until the 2θ value reached 0.65° above the 2θ angle at which $K\alpha_2$ was diffracted. Two 10-sec background counts were taken with the apparatus stationary at 0.40° below and above the 2θ angles at which scanning was begun and ended, respectively. Three moderate intensity reflections were remeasured periodically to check for crystal decomposition or machine malfunctions, but no significant variation was observed.

All calculations were done using a CDC 6600 computer. The raw data were converted into intensities, *I*, and standard deviations, $\sigma(I)$, according to the equations

$$I = C - (t_c/2t_b)(B_1 + B_2)$$

and

$$\sigma^2(I) = C + (t_c^2/4t_b^2)(B_1 + B_2)$$

where *C* is the total count accumulated while scanning across a reflection for a time, t_c, and B_1 and B_2 are the background counts obtained in time, t_b. When a reflection was measured more than once, the intensities were averaged and its standard deviation was set equal to the greater of $(1/n)(\Sigma\sigma_i^2)^{1/2}$ or $(1/(n-1))(\Sigma\Delta_i^2)^{1/2}$, where σ_i and Δ_i are the standard deviation of the *i*th measurement and the deviation of the *i*th measurement from the average, respectively, and *n* is the number of measurements of the *i*th reflection. To reduce the weight assigned to the large intensities, an additional term, $(0.03I)^2$, was included in the calculation of $\sigma^2(F^2) = (\mathrm{LP})^2 \cdot [\sigma^2(I) + (0.03I)^2]$.

All except 16 of the *hkl* and $hk\bar{l}$ reflections within the sphere of reflection bounded by $\sin\theta/\lambda = 0.6216$ ($2\theta_{\max} = 147°$) were measured. Excluding systematic space group absences, there were 3242 independent data, of which 508 had intensities less than the standard deviations, including 232 reflections measured to be zero.

The absorption coefficient for this compound is $\mu = 13.7\ \mathrm{cm}^{-1}$. No correction for absorption effects was applied. It is estimated that absorption effects introduce a maximum error of 8% in the intensity data.

Lorentz and polarization factors were applied and the data were converted to structure factors. Refinements were done using an unpublished version of a full matrix least-squares program, which minimizes the quantity $\Sigma w(|F_o| - |F_c|)^2/\Sigma w|F_o|^2$, where F_o and F_o are the observed and calculated structure factors and *w* is the weighting factor. In the original refinements, $w = 1$ was used for all reflections; but for the final refinements, $w = [\sigma(F)]^{-2}$ was used where $\sigma(F) = [\sigma(F^2)]^{1/2}$, if $I \leqq \sigma(I)$, and $\sigma(F) = F - [F^2 - \sigma(F^2)]^{1/2}$, if $I > \sigma(I)$. Anisotropic temperature factors used have the form $\exp(-\beta_{11}h^2 - \beta_{22}k^2 - \beta_{33}l^2 - 2\beta_{12}hk - 2\beta_{13}hl - 2\beta_{23}kl)$. All atoms were considered to be in their neutral valence states. The scattering factors of Cromer and Waber[8] were used for all atoms except hydrogen whose scattering factors are those of Stewart, Davidson, and Simpson.[9] Cromer's corrections[10] for anomalous dispersion applied to the scattering factors of the nickel are $\Delta f' = -3.20$ and $\Delta f'' = +0.67$ electron. The stereoscopic pair drawings were prepared using Johnson's ORTEP program.[11]

Crystal Data

The crystals are monoclinic with unit cell dimensions, measured at room temperature (~22°), $a = 13.371 \pm 0.003$, $b = 10.398 \pm 0.005$, $c = 13.556 \pm 0.003$ Å, $\beta = 119.16 \pm 0.04°$. There are four molecules of $Ni(B_9C_2H_{11})_2$ in the unit cell. The calculated density, $\rho = 1.31$ g/cc, agrees well with the observed density, $\rho = 1.30$ g/cc, measured by flotation.

The extinction rules found, 0*k*0, $k = 2n + 1$ and *h*0*l*, $l = 2n + 1$, are characteristic of space group $P2_1/c$. All atoms lie on the general symmetry equivalent positions, $\pm(x, y, z$ and $x, \frac{1}{2} - y, \frac{1}{2} + z)$.

Determination of the Structure

There are four general symmetry equivalent positions in this space group and there are four molecules of $Ni(B_9C_2H_{11})_2$ in the unit cell; thus, the asymmetric unit is one complete molecule. Since the pattern of strong and weak intensities was dominated by the nickel atom, its coordinates were readily found by testing trial coordinate values in the structure factor equation. The

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(6) M. R. Churchill, K. Gold, J. N. Francis, and M. F. Hawthorne, *J. Amer. Chem. Soc.*, **91**, 1222 (1969).

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(11) C. K. Johnson, "ORTEP, A Fortran Thermal-Ellipsoid Plot Program for Crystal Structure Illustrations," Report ORNL-3794, Oak Ridge National Laboratory, Oak Ridge, Tenn., 1965.

Table I. Positional Parameters ($\times 10^5$) and Thermal Parameters ($\times 10^2$) for all Nonhydrogen Atoms[a-c]

Atom	x	y	z	B_{11}	B_{22}	B_{33}	B_{12}	B_{13}	B_{23}
Ni	25452 (2)	06001 (3)	25232 (2)	204 (1)	196 (1)	186 (1)	−03 (1)	98 (1)	−09 (1)
Atoms in cage 1									
C(1)	26879 (15)	25130 (17)	21672 (15)	314 (7)	222 (7)	237 (7)	−01 (6)	162 (6)	09 (6)
C(2)	13990 (15)	20935 (19)	17896 (15)	259 (7)	285 (9)	218 (7)	40 (6)	99 (6)	−16 (6)
B(4)	36258 (18)	20898 (21)	35311 (18)	245 (8)	246 (9)	245 (8)	−22 (7)	106 (7)	−22 (7)
B(5)	31486 (19)	37147 (23)	31314 (19)	341 (10)	223 (10)	306 (9)	−20 (8)	154 (8)	−37 (7)
B(6)	17313 (20)	37024 (23)	19900 (19)	383 (10)	251 (10)	280 (9)	71 (8)	148 (8)	06 (8)
B(7)	12810 (17)	13113 (22)	28568 (18)	244 (8)	303 (9)	289 (9)	04 (7)	156 (7)	−25 (7)
B(8)	27455 (18)	13394 (22)	40670 (17)	304 (8)	290 (10)	198 (8)	04 (7)	136 (7)	−11 (7)
B(9)	31460 (19)	30034 (22)	43161 (18)	322 (9)	283 (10)	232 (8)	05 (8)	103 (7)	−56 (7)
B(10)	19789 (20)	39795 (23)	33749 (19)	383 (10)	276 (10)	261 (9)	55 (8)	137 (8)	−44 (7)
B(11)	08508 (18)	29553 (23)	24523 (18)	273 (9)	344 (11)	276 (9)	77 (8)	119 (7)	−45 (8)
B(12)	17116 (19)	25312 (23)	38973 (18)	333 (9)	340 (10)	246 (8)	30 (8)	166 (7)	−41 (8)
Atoms in cage 2									
C(1)	24025 (16)	−03074 (18)	10997 (14)	328 (8)	255 (8)	199 (7)	44 (6)	121 (6)	07 (6)
C(2)	37008 (15)	−02532 (18)	21221 (15)	273 (7)	268 (8)	263 (7)	20 (6)	163 (6)	11 (6)
B(4)	14645 (19)	−08839 (22)	15061 (19)	264 (8)	253 (10)	276 (9)	−40 (7)	94 (7)	−59 (7)
B(5)	19396 (20)	−18226 (24)	07177 (19)	387 (10)	282 (10)	265 (9)	26 (9)	102 (8)	−64 (8)
B(6)	33589 (20)	−13955 (22)	10892 (19)	420 (10)	293 (10)	265 (9)	90 (8)	201 (8)	08 (7)
B(7)	37965 (18)	−07933 (21)	33719 (17)	263 (8)	249 (9)	201 (8)	34 (7)	101 (7)	10 (7)
B(8)	23365 (18)	−12736 (20)	30031 (18)	326 (9)	222 (8)	290 (9)	−22 (7)	186 (8)	17 (7)
B(9)	19171 (22)	−24717 (24)	19148 (21)	410 (11)	232 (10)	386 (11)	−47 (8)	192 (10)	−34 (8)
B(10)	30930 (22)	−27930 (23)	16777 (20)	437 (11)	257 (10)	338 (10)	49 (9)	183 (9)	−21 (8)
B(11)	42256 (18)	−17369 (23)	25368 (18)	306 (9)	286 (9)	292 (9)	80 (8)	158 (7)	21 (8)
B(12)	33653 (21)	−24149 (23)	30686 (19)	399 (10)	238 (10)	300 (9)	47 (8)	187 (8)	49 (8)

[a] The numbering system is shown in Figure 1. [b] Errors estimated by least squares for the least significant digits are given in parentheses following the parameter. [c] The anisotropic thermal parameters, B, in units of Å^2, are given by $B_{ij} = 4\beta_{ij}/a_i^*a_j^*$, where a_i^* is the length of the ith reciprocal cell dimension.

Table II. Hydrogen Positional Parameters ($\times 10^4$) and Thermal Parameters ($\times 10$)[a,b]

Atom	x	y	z	B
Atoms in cage 1				
H(1)	2916 (14)	2507 (16)	1632 (14)	22 (4)
H(2)	0962 (15)	1870 (20)	1037 (16)	32 (4)
H(4)	4509 (15)	1890 (18)	3779 (15)	30 (4)
H(5)	3763 (16)	4379 (17)	3133 (15)	29 (4)
H(6)	1440 (15)	4284 (17)	1268 (15)	27 (4)
H(7)	0571 (15)	0643 (16)	2679 (15)	30 (4)
H(8)	3071 (16)	0664 (17)	4780 (16)	33 (4)
H(9)	3748 (16)	3302 (19)	5189 (16)	38 (4)
H(10)	1826 (15)	4912 (20)	3635 (15)	31 (4)
H(11)	−0046 (15)	3131 (18)	2033 (15)	31 (4)
H(12)	1341 (16)	2537 (18)	4468 (15)	36 (4)
Atoms in cage 2				
H(1)	2193 (16)	0348 (18)	0554 (16)	31 (4)
H(2)	4163 (16)	0451 (18)	2099 (16)	34 (5)
H(4)	0619 (17)	−0569 (18)	1087 (17)	38 (5)
H(5)	1340 (16)	−1983 (20)	−0192 (17)	41 (5)
H(6)	3670 (16)	−1256 (19)	0486 (16)	40 (5)
H(7)	4462 (16)	−0493 (17)	4125 (15)	29 (4)
H(8)	2015 (15)	−1369 (18)	3580 (15)	30 (4)
H(9)	1300 (17)	−3179 (21)	1842 (17)	43 (5)
H(10)	3260 (17)	−3753 (20)	1466 (17)	40 (5)
H(11)	5148 (15)	−1888 (17)	2882 (15)	28 (4)
H(12)	3679 (15)	−3132 (19)	3721 (15)	32 (4)

[a] The number in the atom label refers to the number of the position in Figure 1 of the atom to which the hydrogen is bonded. [b] Errors estimated by least squares for the least significant digits are given in parentheses following the parameter.

conventional R value, $R = \Sigma||F_o| - |F_c||/\Sigma|F_o|$, based on a least-squares refinement of an overall scale factor and three coordinates and an isotropic thermal parameter for the nickel, stopped at $R = 0.46$. A Fourier synthesis using phases calculated from the nickel atom only, contained peaks, some rather poorly resolved, corresponding to the 22 carbon and boron atoms. When these atoms, all with the scattering power of boron, were added to the refinement, the R value dropped to 0.14. Thermal parameters and bond distances gave exceptionally clear indications of the positions of the two carbon atoms in each cage, and in all further refinements, these atoms were given the scattering power of carbon. When some blunders in the intensity data were removed and when the anomalous dispersion corrections were applied, full-matrix least-squares refinement of a scale factor, three positional coordinates, and six anisotropic thermal parameters for each of the 23 atoms gave $R = 0.086$ for all 3242 data. On a difference Fourier using phases calculated from the atoms in this refinement appeared all of the 22 hydrogen atoms. When three positional parameters and an isotropic thermal parameter for each hydrogen were included, and when weighting factors derived from the σ's were used, full-matrix least-squares refinement gave $R = 0.072$ for all 3242 data. It was noticed that all of the five maximum intensity reflections were observed much too low, suggesting that their counting rates exceeded the capacity of the scintillation counter; therefore, they were given zero weight in all further least-squares refinement. The final series of refinement was carried out on an overall scale factor, 3 positional parameters and 6 anisotropic thermal parameters for each of the 23 nonhydrogen atoms and 3 positional parameters and an isotropic thermal parameter for each of the 22 hydrogen atoms. The 5 maximum intensity reflections and all reflections for which $I < \sigma(I)$ were given zero weight. This refinement gave the conventional R values of $R = 0.035$ for the 2724 nonzero-weighted data, and $R = 0.051$ calculated using all 3242 data. The weighted R value, $R_2 = [\Sigma w(|F_o| - |F_c|)^2/\Sigma w|F_o|^2]^{1/2}$, reached $R_2 = 0.031$. The standard deviation of observation of unit weight, given by $[\Sigma w(|F_o| - |F_c|)^2/(n - p)]^{1/2}$, where n and p are the number of data and the number of parameters, respectively, was 1.2. Root mean

Table III. Observed and Calculated Structure Factors[a]

L	FOB	FCA
H,K= 0, 0		
2	646	748*
4	113	114
6	159	156
8	76	73
10	53	54
12	85	84
14	59	59
H,K= 0, 1		
1	105	105
2	229	227
3	24	24
4	133	132
5	65	63
6	96	89
7	26	25
8	0	4*
9	12	12
10	16	19
11	10	12
12	13	14
13	19	19
14	13	15
H,K= 0, 2		
0	150	143
1	90	84
2	61	57
3	143	145
4	128	126
5	147	148
6	124	126
7	122	125
8	138	140
9	92	91
10	73	70
11	41	40
12	32	32
13	37	37
14	28	29
H,K= 0, 3		
1	9	7
2	50	51
3	120	114
4	82	80
5	58	55
6	44	43
7	22	19
8	48	47
9	55	57
10	39	37
11	13	16
12	7	4*
13	14	11
14	17	17
H,K= 0, 4		
0	41	43
1	132	135
2	35	36
3	241	242
4	27	26
5	318	330
6	23	20
7	183	187
8	16	17
9	92	93
10	0	4*
11	86	86
12	5	11*
13	43	45
H,K= 0, 5		
1	60	58
2	3	6*
3	17	16
4	24	23
5	4	4*
6	8	8*
7	78	80
8	6	2*
9	22	24
10	30	31
11	16	18
12	7	3*
13	11	11
H,K= 0, 6		
0	151	154
1	149	151
2	150	152
3	135	136
4	57	57
5	6	6*
6	13	12
7	9	7*
8	36	36
9	61	62
10	48	50
11	46	45
12	35	34
H,K= 0, 7		
1	5	2*
2	6	7*
3	15	16
4	0	2*
5	7	2*
6	37	39
7	33	36
8	43	45
9	9	2
10	17	14
11	16	15
12	11	12
H,K= 0, 8		
0	50	51
1	7	9*
2	53	52
3	9	14
4	101	101
5	17	18
6	106	108
7	0	1*
8	101	100
9	19	17
10	127	125
11	30	30
H,K= 0, 9		
1	4	7*
2	20	23
3	6	0*
4	2	1*
5	9	8
6	24	24
7	4	5*
8	24	22
9	5	2*
10	12	12
H,K= 0, 10		
0	73	73
1	53	52
2	76	75
3	61	61
4	69	69
5	44	44
6	70	69
7	32	33
8	42	43
9	5	6*
H,K= 0, 11		
1	20	10
2	28	26
3	22	20
4	7	9*
5	16	16
6	5	3*
7	19	18
H,K= 0, 12		
0	32	33
1	73	71
2	13	14
3	44	44
4	0	3*
5	51	52
H,K= 1, 0		
-14	23	23
-12	13	11
-10	18	20
-8	22	21
-6	1	9*
-4	25	26
-2	24	25
0	44	41
2	13	15
4	21	21
6	0	4*
8	5	8*
10	0	1*
12	22	20
14	16	14
H,K= 1, 1		
-15	46	46
-14	10	11
-13	78	77
-12	19	22
-11	55	55
-10	25	25
-9	69	69
-8	47	47
-7	145	146
-6	92	91
-5	164	160
-4	106	104
-3	167	165
-2	152	152
-1	438	457*
0	201	206
1	365	377*
2	91	93
3	146	149
4	91	92
5	235	238
6	101	101
7	230	235
8	63	65
9	130	131
10	39	40
11	58	57
12	23	25
13	36	36
14	13	12
H,K= 1, 2		
-14	12	14
-13	13	15
-12	10	14
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8	149	147
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H,K= 2, 3		
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H,K= 2, 7		
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H,K= 2, 9		
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H,K= 2, 10		
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H,K= 3, 11		
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6	120	121
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7	26	23
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H,K= 4, 7		
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-1	23	23
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2	63	63
3	68	68
4	53	54
5	59	61
6	41	42
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-7	0	10*
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-1	37	37
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3	18	18
4	19	20
5	8	2
6	8	6
H,K= 8, 8		
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-6	62	60
-5	14	14
-4	79	77
-3	14	8
-2	79	81
-1	7	7*
0	73	74
1	0	3*
2	72	73
3	11	12
4	9	7
5	0	0*
H,K= 8, 9		
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-5	6	12*
-4	6	5*
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1	11	14
2	36	36
3	14	16
H,K= 8, 10		
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-8	23	25
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-5	38	37
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0	32	34
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H,K= 9, 0		
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-6	5	7*
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4	15	15
6	18	20
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-8	36	36
-7	87	87
-6	19	12
-5	53	54
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3	73	72
4	37	36
5	52	52
6	19	23
7	37	37
H,K= 9, 2		
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-3	49	50
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0	24	24
1	41	44
2	8	3*
3	20	23
4	15	11
5	22	23
6	13	11
7	19	19
H,K= 9, 3		
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-15	21	20
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-11	42	43
-10	98	97
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-7	85	85
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-4	135	132
-3	38	35
-2	42	39
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0	15	13
1	22	26
2	56	57
3	26	25
4	97	95
5	35	33
6	76	75
7	44	44
H,K= 9, 4		
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-7	16	10
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-2	20	26
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0	22	23
1	8	12*
2	23	24
3	35	33
4	21	20
5	23	25
6	9	5
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-13	12	15
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-9	18	20
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3	10	11
4	45	47
5	7	8*
6	18	19

Table III (*Continued*)

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-6	9	10*
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-4	12	14
-3	26	25
-2	28	25
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0	42	42
1	16	16
2	15	17
3	20	18
4	0	10*
5	4	4*
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-5	66	66
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-3	58	55
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-1	73	73
0	41	39
1	81	81
2	35	36
3	42	41
4	6	2*
H,K=	9,	8
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-9	25	21
-8	0	4*
-7	0	9*
-6	0	2*
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-4	21	23
-3	8	13*
-2	22	20
-1	20	19
0	0	11*
1	12	6
2	19	23
3	6	8*
H,K=	9,	9
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0	19	17
1	11	4
2	12	14
H,K=	9,	10
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4	71	70
6	93	93
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-9	26	28
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1	12	13
2	13	17
3	0	5*
4	5	7*
5	23	20
6	5	6*
H,K=	10,	2
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-15	19	18
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0	83	81
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3	38	39
4	46	44
5	31	32
6	37	36
H,K=	10,	3
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3	49	49
4	21	19
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H,K=	10,	5
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3	19	21
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5	20	19
H,K=	10,	6
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2	26	23
3	29	31
4	28	27
H,K=	10,	7
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H,K=	10,	8
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H,K=	10,	9
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1	56	57
2	5	11*
3	35	34
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3	14	11
4	15	16
5	6	4*
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4	67	66
H,K=	11,	4
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1	38	35
2	56	56
3	33	35
4	34	32
H,K=	11,	5
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2	16	15
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2	0	10*
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H,K=	12,	3
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3	68	69
H,K=	12,	5
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H,K=	12,	7
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H,K=	12,	8
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H,K=	13,	0
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H,K=	13,	1
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0	12	12
1	20	19
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-10	15	12
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H,K=	13,	5
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0	37	35
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-3	13	11
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H,K=	14,	3
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-1	26	26
0	46	46
H,K=	14,	4
-13	34	35
-12	11	9
-11	53	53
-10	9	5
-9	36	37
-8	0	11*
-7	27	26
-6	21	18
-5	46	46
-4	0	4*
-3	38	38
-2	8	9
-1	42	43
H,K=	14,	5
-12	25	24
-11	44	45
-10	21	19
-9	4	1*
-8	11	10
-7	27	26
-6	19	20
-5	19	17
-4	20	19
-3	4	4*
-2	11	12
H,K=	14,	6
-10	8	7
-9	6	8*
-8	15	14
-7	23	20
-6	26	25
-5	35	36
-4	22	25
H,K=	15,	0
-12	12	9
-10	16	13
-8	7	13*
-6	16	14
-4	13	10
-2	12	6
H,K=	15,	1
-13	35	36
-12	0	3*
-11	48	48
-10	12	11
-9	75	74
-8	31	30
-7	69	69
-6	30	30
-5	47	47
-4	15	12
-3	39	39
-2	5	7*
H,K=	15,	2
-13	11	12
-12	20	21
-11	18	18
-10	48	47
-9	25	26
-8	51	51
-7	11	2
-6	15	14
-5	20	21
-4	9	9
-3	41	41
-2	6	2*
H,K=	15,	3
-12	35	37
-11	19	18
-10	46	46
-9	16	13
-8	42	44
-7	16	20
-6	37	36
-5	20	21
-4	68	67
-3	28	29
H,K=	15,	4
-11	6	2*
-10	8	3
-9	1	3*
-8	28	29
-7	16	10
-6	6	8*
-5	18	17
-4	38	39
H,K=	15,	5
-9	7	6
-8	12	12
-7	2	1*
-6	35	35
-5	7	6
H,K=	16,	0
-10	33	33
-8	24	24
-6	32	33
H,K=	16,	1
-11	14	11
-10	10	11
-9	18	18
-8	6	5*
-7	19	22
-6	9	5
-5	3	7*
H,K=	16,	2
-10	30	31
-9	55	55
-8	52	51
-7	44	44
-6	34	35
-5	23	22
H,K=	16,	3
-9	41	41
-8	10	4
-7	7	7*

[a] Each datum given zero weight in the final refinement is indicated with an asterisk.

square averages of $(|F_o| - |F_c|)/\sigma(F)$ taken over small intensity intervals approximated this value of 1.2 over the entire intensity range. No parameter shifted by more than 5% of its estimated standard deviation in the final cycle of refinement.

Results and Discussion

Final values for atomic positions and thermal parameters are given in Table I for all the nonhydrogen atoms and in Table II for the hydrogen atoms. The observed and calculated structure factors are shown in Table III.

As shown by the stereoscopic pair drawing in Figure 2, this neutral molecule is a double-cage carborane having the nickel atom sandwiched between the cages by bonding to the open fivefold faces of the two icosahedral fragments. Each of the carbon and boron atoms in the cages has a hydrogen atom pointing out radially from the cage approximately along a line from the atom to which it is attached to the atom directly opposite it in the cage. The carbon pairs in the cages on opposite sides of the metal are as close as is possible with the cages staggered. The carbons allow only C_2 point symmetry for the molecule. This lack of reflection and inversion symmetry permits the existence of enantiomorphic rotational isomers.

An interesting feature of the structure may be seen by passing four planes through the molecule, each containing five atoms. Label the plane through atoms B(5), B(6), B(9), B(11), and B(12) of cage 1 as plane 1, and the plane through the same atoms of cage 2 as plane 4. Let atoms C(1), C(2), B(4), B(7), and B(8) of cage 1 form plane 2, and let the same atoms in cage 2 form plane 3. Then, planes 2 and 3 are tipped from a parallel arrangement in such a way that the distance between the carbon pairs on opposite sides of the nickel is increased. Thus, the distance from C(1) in cage 1 to C(1) in cage 2 is 3.21 Å, while the distance from B(8) in cage 1 to B(8) in cage 2 is 3.00 Å. The vector from Ni to B(10) of cage 1 is parallel to the corresponding vector, Ni to B(10) in cage 2, so the molecule as a whole is not bent. But, taking the best least-squares planes through the atoms, plane 2 makes an angle of 6° with plane 3 and plane 1 makes an angle of 3° with plane 4. The atoms assigned to

Table IV. Bond Distances (Å)[a] Excluding Hydrogen

Atom 1	Atom 2	Cage 1	Cage 2
Ni	C(1)	2.077	2.072
Ni	C(2)	2.065	2.071
Ni	B(4)	2.105	2.104
Ni	B(7)	2.086	2.085
Ni	B(8)	2.120	2.115
C(1)	C(2)	1.601	1.610
C(1)	B(4)	1.710	1.706
C(1)	B(5)	1.692	1.680
C(1)	B(6)	1.710	1.713
C(2)	B(6)	1.718	1.720
C(2)	B(7)	1.732	1.729
C(2)	B(11)	1.670	1.675
B(4)	B(5)	1.794	1.778
B(4)	B(8)	1.832	1.827
B(4)	B(9)	1.764	1.753
B(7)	B(8)	1.843	1.833
B(7)	B(11)	1.802	1.791
B(7)	B(12)	1.772	1.765
B(8)	B(9)	1.794	1.799
B(8)	B(12)	1.786	1.786
B(5)	B(6)	1.769	1.767
B(5)	B(9)	1.770	1.771
B(6)	B(11)	1.761	1.762
B(9)	B(12)	1.783	1.802
B(11)	B(12)	1.777	1.774
B(10)	B(5)	1.770	1.770
B(10)	B(6)	1.764	1.775
B(10)	B(9)	1.776	1.783
B(10)	B(11)	1.771	1.771
B(10)	B(12)	1.773	1.781

[a] Standard deviations calculated from the least-squares estimates of the errors in coordinates are the following: ±0.002 Å on all distances to Ni; ±0.002 on C(1)–C(2) in each cage; ±0.004 on B(9)–B(10) and B(9)–B(12) in cage 2; and ±0.003 on all others.

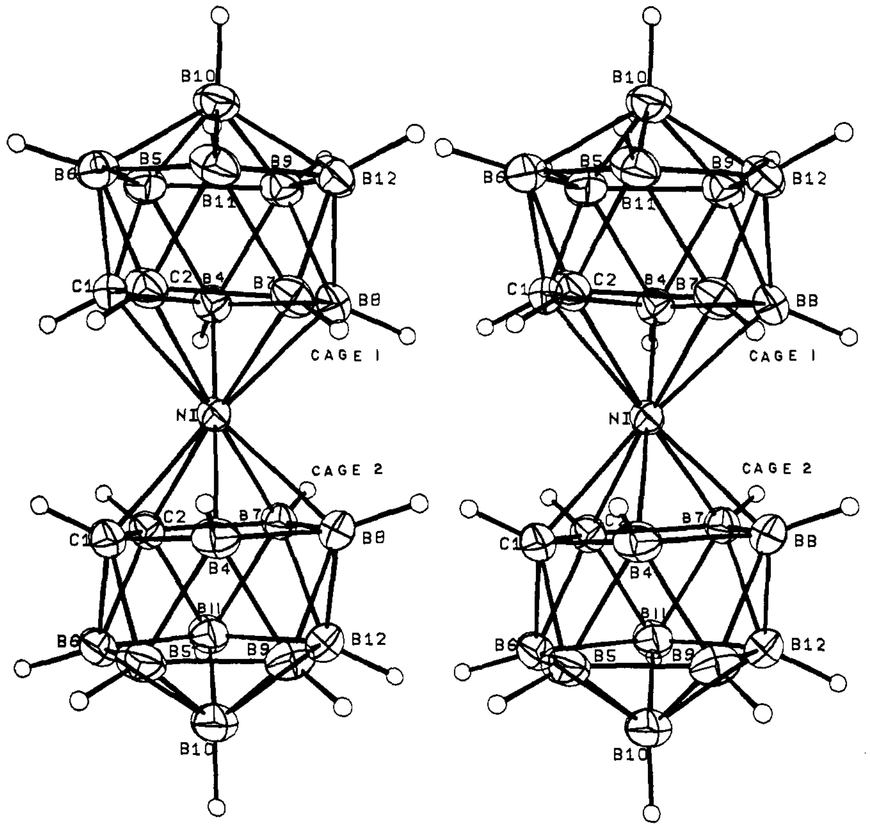

Figure 2. Stereoscopic pair drawing of a complete $Ni(B_9C_2H_{11})_2$ molecule. Thermal ellipsoid boundaries are at the 40% probability level. For this drawing the hydrogens were given artificial thermal parameters, $B = 0.5$.

planes 2 and 3, however, are not exactly planar. If only the three boron atoms B(4), B(7), and B(8) are included in the calculation of these planes, the angle between them is only 2°. The carbon atoms are displaced out of these planes toward B(6) by 0.09 Å in cage 1 and by 0.07 Å in cage 2. Thus, the carbon atoms in each cage are displaced approximately toward the geometric center of each 12-membered cage, up toward B(6), but in toward the center until the C–Ni distances are less than the B–Ni distances. That planes 1 and 4 are not parallel is probably due to shifts of the atoms in these planes, especially B(6), as compensation for the displacements of the carbon atoms.

Interatomic distances are given for all atoms except hydrogens in Table IV. The distances between the hydrogen atoms and the carbons or borons to which they are attached are given in Table V. Since the asymmetric unit consists of both cages of one molecule, for every distance in one cage there is another independent value of an equivalent distance in the other cage. Having two values for every type of bond distance allows one to calculate average estimated errors based on the discrepancies between each pair of distances by the equation $\sigma = (\Sigma\Delta_i^2/n)^{1/2}$, where Δ_i is the difference between the bond distance in one cage and the corresponding bond distance in the other cage, and where n is the number of pairs. This gives $\sigma = \pm 0.004$ Å on the Ni to C or B distances, $\sigma = \pm 0.008$ on distances involving only carbons and borons, and $\sigma = \pm 0.04$ on the distances between a hydrogen and the carbon or boron to which it is attached. These rms estimates of error based on the scatter are all larger than the errors calculated from the errors in coordinates estimated from the least-squares refinements ($\sigma = \pm 0.002$, ±0.002, and ±0.02 Å, respectively), and it is the opinion of the authors that the rms errors are more realistic estimates of the actual errors in these interatomic distances. The intramolecular distances between the hydrogen atoms of cage 1 and those of cage 2 fall into four groups: 2.59 Å for H(1) to H(1)′ and H(2)′, for H(2) and H(1)′ and H(4)′, and for H(4) to H(2)′; 2.53 Å for H(4) to H(7)′ and for H(7) to H(4)′; 2.70 Å for H(7) to H(8)′ and for H(8) to H(7)′; and 2.62 Å for H(8) to H(8)′. The unprimed and primed labels refer to atoms in cage 1 and cage 2, respectively. All standard deviations are ±0.03 Å.

Averages of various types of bond angles are given in Table VI. There are no surprises in the table since the angles differ, as expected, from the values of 60 and 108° for the angles in the triangular and pentagonal

Table V. Bond Distances (Å) between an Atom and Its Hydrogen[a]

Atom	Cage 1	Cage 2
C(1)	0.91	0.94
C(2)	0.93	0.97
B(4)	1.08	1.04
B(5)	1.07	1.11
B(6)	1.05	1.09
B(7)	1.11	1.02
B(8)	1.10	1.07
B(9)	1.10	1.07
B(10)	1.09	1.09
B(11)	1.06	1.10
B(12)	1.10	1.07

[a] Standard deviations calculated from least-squares estimates of the errors in coordinates are ±0.02 Å in all distances.

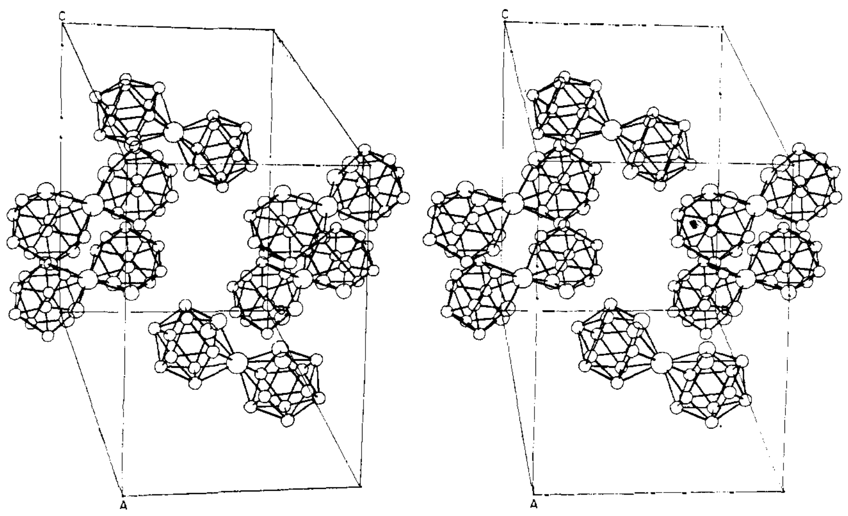

Figure 3. Stereoscopic pair drawing of the unit cell (hydrogen atoms not shown). The origin is at the left rear corner of the unit cell. All thermal ellipsoids here are artificial. Nickel atoms are shown as the largest circles, medium size circles designate carbon atoms, and boron atoms are shown by the smallest circles.

rings, respectively, of a regular icosahedron distorted by insertion of two carbons and a nickel atom into the cage.

Table VI. Average Bond Angles[a]

Type				
Atom 1	Atom 2	Atom 3	No.	Angle, deg
Angles around all triangular faces				
C	Ni	C	2	45.7 ± 0.2
C	Ni	B	4	48.7 ± 0.3
B	Ni	B	4	51.6 ± 0.2
Ni	C	C	4	67.2 ± 0.3
Ni	C	B	4	66.4 ± 0.3
Ni	B	C	4	64.9 ± 0.1
Ni	B	B	8	64.2 ± 0.3
C	B	C	2	55.9 ± 0.2
C	C	B	4	62.1 ± 0.2
B	C	B	8	63.1 ± 0.2
C	B	B	16	58.4 ± 0.3
B	B	B	60	60.0 ± 0.1
Angles around all pentagonal rings				
Ni	C	B	8	124.8 ± 0.3
Ni	B	B	12	116.2 ± 0.4
C	Ni	B	8	83.9 ± 0.9
B	Ni	B	2	88.1 ± 0.4
C	C	B	8	112.1 ± 0.3
C	B	B	24	103.7 ± 0.2
B	B	B	42	108.5 ± 0.3
Angles involving hydrogen atoms attached to atom 2				
Ni	C	H	4	105.4 ± 0.5
Ni	B	H	6	114.3 ± 1.4
C	C	H	4	116.1 ± 0.7
B	C	H	12	118.9 ± 1.1
C	B	H	12	118.6 ± 0.4
B	B	H	72	122.6 ± 0.5

[a] The error of each average is estimated from the scatter of the individual values.

The molecular packing is shown by the stereoscopic pair drawing of the unit cell shown in Figure 3. There are only six intermolecular hydrogen to hydrogen distances less than 2.60 Å. These are H(11)–H(8)′, 2.40 Å; H(10)–H(1)′, 2.42 Å; H(1)–H(11)′, 2.42 Å; H(2)–H(12), 2.49 Å; H(11)–H(5)′, 2.54 Å; and H(12)–H(1)′, 2.58 Å, where the unprimed and the primed labels indicate atoms in cage 1 and cage 2, respectively. Because the center of gravity of the electron density around a bonded hydrogen found in an X-ray analysis is not usually coincident with the position of the nucleus, a hydrogen appears to be too close to the atom to which it is bonded, thus making the intermolecular hydrogen distances appear to be too long. So although the distances appear to be too long for normal van der Waals contacts (2.40 Å), it is likely that they actually are shorter and that normal van der Waals interactions are holding the crystal together.

An indication of the asymmetry of the molecule is demonstrated by the molecular packing in the crystal lattice. The molecule shown in Figure 1 is the particular rotational isomer in which, looking from B(10) in one cage to the nickel, the opposite cage is staggered by a clockwise rotation of 36° from the position in which the carbons would be eclipsed. An equally probable isomer should and does result from a counterclockwise rotation from the eclipsed position. In a more symmetrical molecule, these two isomers might be sufficiently equivalent in shape that either isomer could pack in any given position in the crystal lattice. If this were the case, C(2) would appear to be equally disordered with B(4) in both cages. In this work, however, it is found that both isomers are present in the crystal and that they are sufficiently different that each must occupy its own position in the lattice. The two enantiomorphic rotational isomers are related to each other here by the *c* glide plane and the inversion center.

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