Table II. Elemental Analysis of $Co(O_3PC_6H_5)(NH_3)$

	% C	% H	% N
actual	31.11	3.40	5.80
theoret	31.05	3.48	6.05

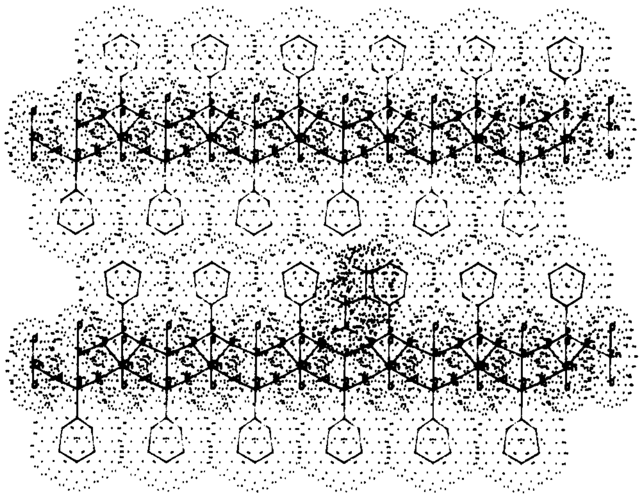

Figure 5. Computer simulation of zinc phenylphosphonate with intercalated propylamine generated with an E&S PS-390 graphics system. Dots indicate extent of van der Waals interactions.

vapor has taken place. This same behavior was observed with $Zn(O_3PC_6H_5)(NH_3)$.

Further evidence for the formation of $Co(O_3PC_6H_5)(NH_3)$ can be revealed through thermogravimetric analysis, elemental analysis, and X-ray powder diffraction. Examination of Figure 4 reveals that loss of NH_3 begins at approximately 110 °C, slightly higher than the onset of water loss in the hydrated cobalt phenylphosphonate. The loss of mass between 110–300 °C constitutes 7.27% of the total mass. The theoretical value calculated by assuming the formula $Co(O_3PC_6H_5)(NH_3)$ is 7.34%, indicating an excellent agreement with the expected value. Further support of successful occupation of the empty coordination site of dehydrated cobalt phenylphosphonate by ammonia is the elemental analysis of $Co(O_3PC_6H_5)(NH_3)$. Table II shows good agreement between the theoretical and actual values. The slight decrease in the percent N found relative to the theoretical value may be due to the incomplete occupation of all the empty coordination sites. Furthermore, the powder pattern of $Co(O_3PC_6H_5)(NH_3)$ shows an interlayer (010) spacing of 14.2 Å, which is consistent with the value observed for the hydrated compound.

In conclusion, the phenylphosphonates were able to readily intercalate ammonia but not larger molecules as does the methyl derivative. Neither two nor three atom molecules such as ethylene, oxygen, or carbon dioxide were intercalated. While it may be argued that these molecules are much less basic than ammonia, they also appear to be restricted by the positioning of the phenyl groups. Computer-simulated structures indicate that the phenyl ring protons provide a barrier to positioning of small molecules at the open coordination site. This is graphically illustrated by the positioning of propylamine at the vacant coordination site of zinc phenylphosphonate (Figure 5). It can be seen that there is significant overlap of the van der Waals surfaces. Even when the rings on alternate sites are turned 90° to each other, the same interference occurs. This is also the case when CO is substituted for propylamine. Therefore, additional synthetic procedures are in progress to alter structures to allow accessibility to the metal site.

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Inclusion of a Small Molecule in a Big Cage: Preparation and Structure of *catena*-[*catena*-(α,ω-Diaminooctane)cadmium-μ-tetracyanonickelate]-Toluene(1/1)

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The preparation and crystal structure of the title compound are described. This compound, $Cd[NH_2(CH_2)_8NH_2]Ni(CN)_4 \cdot C_6H_5CH_3$, crystallizes in the monoclinic space group $P2/m$ with $a = 11.348$ (3) Å, $b = 7.652$ (2) Å, $c = 7.042$ (1) Å, $\alpha = 90.00°$, $\beta = 106.07$ (2)°, $\gamma = 90.00°$, and $Z = 1$. The structure determination indicated the inclusion of a toluene molecule, which is orientationally disordered within a rigid host cage. The distribution of orientations was modeled by analyzing the occupancies of the toluene guest atoms.

Introduction

Crystal inclusion chemistry has recently attracted interest in a variety of fields.[1,2] Since K. A. Hofmann reported the synthesis of $Ni(CN)_2 \cdot NH_3 \cdot C_6H_6$,[3] several compounds of this type have been investigated extensively and can be categorized as classical clathrates.[4-6] After Iwamoto replaced a pair of ammine groups by ethylenediamine,[7] both the Iwamoto group[8] and the Davies group[9] have subsequently reported that a long α,ω-diaminoalkane can replace a pair of ammine groups in the clathrate series to give a novel three-dimensional lattice-structure compound (Hofmann-diam-type): $Cd[NH_2(CH_2)_nNH_2]Ni(CN)_4 \cdot Guest$.[10-14] In the case where n is even, the typical structure consists of ridged cyanometal sheets bridged by all-*trans*-diaminoalkanes. The cage formed by this lattice can contain aromatic guest molecules. We

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(1) Atwood, J. L.; Davies, J. E. D.; MacNicol, K. K., Eds. *Inclusion Compounds*; Academic Press: London, 1984; Vol. 1.
(2) For example: Miki, K.; Masui, A.; Miyata, M.; Shibakami, N.; Takemoto, K. *J. Am. Chem. Soc.* **1988**, *90*, 6594 and references therein.
(3) Hofmann, K. A.; Kuspert, Z. *Z. Anorg. Chem.* **1897**, *15*, 204.
(4) Iwamoto, T. The Hofmann-Type and Related Inclusion Compounds. In ref 1, pp 29–57.
(5) Sharpe, A. G. *The Chemistry of Cyanocomplexes of the Transition Metals*; Academic Press: London, 1976.
(6) Cernak, J.; Dunaj-Jurco, M.; Melnik, M.; Chomic, J.; Skorsepa, J. *Rev. Inorg. Chem.* **1988**, *9*, 259.
(7) Iwamoto, T. *Inorg. Chim. Acta* **1968**, *2*, 269.
(8) Hasegawa, T.; Nishikiori, S.; Iwamoto, T. *J. Inclusion Phenom.* **1984**, *1*, 365.
(9) Davies, J. E. D.; Marer, A. M. *J. Mol. Struct.* **1983**, *102*, 203.

Table I. Crystal Data

formula	$Cd[NH_2(CH_2)_8NH_2]$- $Ni(CN)_4 \cdot C_6H_5CH_3$	V, Å^3	587.7
		Z	1
fw	511.59	ρ_{calc}, g cm^{-3}	1.458
space group	$P2/m$	T, K	298
a, Å	11.348 (3)	λ, Å	0.71069
b, Å	7.652 (2)	μ(Mo Kα), cm^{-1}	17.4
c, Å	7.042 (1)	$R = R(F_o)$	0.025
β, deg	106.07 (2)	$R_w = R_w(F_o)$	0.035

can compare the cyanometal sheets, the diaminoalkanes, and the guest molecules to the floors, the walls, and the tenants, respectively, of an apartment building. Hence, we shall call this series of clathrates molecular apartments (MA) and abbreviate them as MA$_n$–xG[15] in this paper.

Of the MAs with well-defined crystal structures,[10,13] the MA$_6$ host is currently the largest cage structure that accommodates an aromatic molecule.[16] The three toluidine isomers and 2,4-xylidine are each suitable guests in a MA$_6$ cavity. If we can synthesize a MA consisting of a larger host and a smaller guest than in MA$_6$–toluidine while preserving the host's structural motif, it is likely that the MA structure is determined primarily by the host structure and is independent of the nature of the small guests. This also suggests that we may use a MA clathrate as a stable molecular vessel to investigate the character of a guest molecule. Motivated by these ideas, we describe here the synthesis and the structure of MA$_8$–toluene, which includes both the longest α,ω-diaminoalkane and the smallest aromatic molecule in the MA series so far.

Experimental Section

Potassium tetracyanonickelate was prepared by conventional methods[17] using potassium cyanide (J. T. Baker Chemical Co.) and nickel(II) chloride hexahydrate (J. T. Baker Chemical Co.) and recrystallized from water. Citric acid (Sigma Chemical Co.), cadmium chloride hydrate (Allied Chemical & Dye Corp.) and all other chemicals (Aldrich Chemical Co.) were used without further purification.

Both $CdCl_2 \cdot 2.5H_2O$ (9 g, 40 mmol) and $K_2[Ni(CN)_4] \cdot H_2O$ (10.5 g, 40 mmol) were dissolved in 400 mL of H_2O. $NH_2(CH_2)_8NH_2$ (115 g, 80 mmol) was added to the solution to form a creamy suspension. Then citric acid (28 g) and 2-aminoethanol (30 mL) were added, and the mixture was stirred vigorously to dissolve the diaminooctane completely. After filtration with a Millipore filter (pore size = 0.45 μm), a golden yellowish solution at pH 9.66 was obtained. About 20 mL of the filtrate was transferred into a vial, and neat toluene (10 mL) was placed over the solution. The vial was left under ambient temperature (~25 °C) for approximately 1 month. Single crystals were formed at the interface and the bottom. Anal. Found: C, 44.48; H, 5.60; N, 16.43; Cd, 21.7; Ni, 11.3. Calcd for $C_{19}H_{28}N_6CdNi$: C, 44.61; H, 5.51; N, 16.43; Cd, 21.97; Ni, 11.48.

In some cases, a coordination compound formed instead of the clathrate compound. This compound is thought to be $Cd[NH_2(CH_2)_8NH_3]_2[Ni(CN)_4]_2$. Its formation was independent of pH, temperature, and concentration. Further investigation revealed that an impurity in toluene catalyzes the formation of this coordination compound. Anal. Found: C, 39.58; H, 5.85; N, 22.94; Cd, 15.20; Ni, 15.81. Calcd for $CdNi_2C_{24}H_{42}N_{12}$: C, 39.57; H, 5.81; N, 23.07; Cd, 15.43; Ni, 16.12. Its structure is now under investigation.

A single crystal of dimensions 0.15 × 0.2 × 0.25 mm was coated with epoxy resin in order to reduce decomposition and evaporation of the guest molecule. Data was collected on a Nicolet P2$_1$ automated four-circle diffractometer using graphite-monochromatized Mo Kα radiation. All calculations were carried out on a VAXstation II computer using the Enraf-Nonius SDP program package.

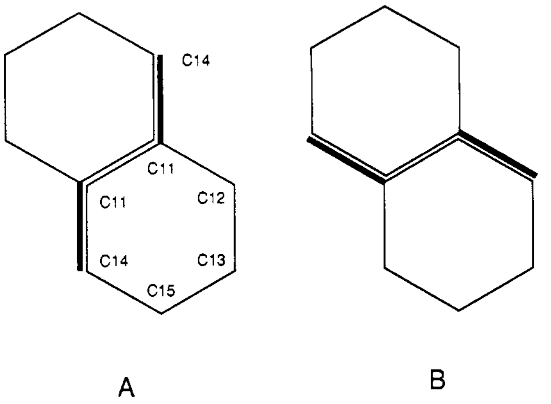

Figure 1. Schematic representation of the possible orientations for toluene within the host structure in $P2/m$.

The crystal and related data are shown in Table I: $CdNiC_{27}H_{44}N_6$, fw = 511.59, monoclinic, $P2/m$, a = 11.348 (3) Å, b = 7.652 (2) Å, c = 7.042 (1) Å, α = 90.00°, β = 106.07 (2)°, γ = 90.00°, Z = 1, V = 587.7 Å^3, ρ_{obs} = 1.44 g cm^{-3}, ρ_{calc} = 1.458, μ(Mo Kα) = 17.4 cm^{-1}. Reflections measured: 5414 over the entire sphere. Reflections used after averaging Friedel pairs and those related by mirror symmetry with $I > 3\sigma(I)$: 990. $(2\theta)_{max}$ = 55°. Check reflections: (020), (1$\bar{1}$1), (100). The decay during data collection was 2.5, 3.75, and 2.5%, respectively; no correction was applied. R_w = 0.032, and R = 0.025. No absorption corrections were applied. The absorption coefficient was 17.205. Data were collected at room temperature (~25 °C).

The heavy-atom method was used for the structure solution and refinement. The space group was determined to be $P2/m$ on the basis of Laue symmetry and success in refinement. There were no systematic absences. The positions of all of the host atoms could be located in the Patterson map. Initial refinement of the host lattice in the space group $P2/m$ was accomplished by using successive Fourier and difference Fourier syntheses and full-matrix least-squares calculations with isotropic B values.

After the host lattice had been refined by including anisotropic thermal parameters for the non-hydrogen atoms, the electron density in an unbiased difference Fourier map ($F_o - F_c$) failed to reveal the location of the guest atoms unambiguously. Placing guest atoms at the positions of highest electron density and refining without restraints consistently led to structures with extremely distorted geometry for the aromatic ring. In order to explore potential solutions for the location of the guest molecule that would satisfy reasonable criteria for acceptable geometry, the method of restrained least-squares refinement (LSRE)[18] was adopted. The procedure in a restrained refinement differs from that of a standard least-squares calculation in that the merit function to be minimized contains not only a χ^2 term due to the deviation of the calculated structure factors from the observed structure factors but also a series of terms that represent the deviation of selected atoms in the crystallographic model from idealized geometry. The weights associated with these additional terms are chosen to match the variances observed for a particular geometry in crystallographic data bases. The relative weight given to the data determines the stiffness of the restraints. The idealized geometry for the toluene molecule included both bond angles and distances as restraints.

The search for possible orientations of the guest molecule was conducted by first positioning the idealized toluene model at those starting orientations that best matched the electron density observed in the unbiased map and then refining each orientation independently with fixed isotropic B values. It is not possible for a single toluene molecule to be present in the unit cell of a centrosymmetric space group unless it is equivalently distributed between inversion-related orientations. Since Z = 1 and the toluene electron density was exclusively in the y = 0.5 plane, the occupancies of all seven toluene atoms were fixed at 0.25 in order to account for the additional orientation implied by the C_2 symmetry of the crystal. After six cycles of LSRE, one structure was observed to have an R value of 0.033, which was significantly lower than the rest. In this structure, four of the atoms in one orientation of the guest molecule roughly overlap with atoms of the C_2-symmetry-related mate, as illustrated in Figure 1A. This overlap is consistent with the magnitude of the electron densities observed in the unbiased map as well as those observed in subsequent difference Fourier maps calculated for each of

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(10) Hasegawa, T.; Nishikiori, S.; Iwamoto, T. *J. Inclusion Phenom.* **1985**, *2*, 351.

(11) Hasegawa, T.; Nishikiori, S.; Iwamoto, T. *Chem. Lett.* **1985**, 1659.

(12) Hasegawa, T.; Nishikiori, S.; Iwamoto, T. *Chem. Lett.* **1986**, 793.

(13) Hasegawa, T.; Iwamoto, T. *J. Inclusion Phenom.* **1988**, *6*, 143.

(14) Nishikiori, S.; Iwamoto, T. *Inorg. Chem.* **1986**, *25*, 788.

(15) This indicates a molecular apartment (MA) with an alkane length of n that accommodates x guest(s) per unit cell. When the conformation is not all-trans or is unknown, we shall denote such a complex by writing mA$_n$–xG.

(16) Recently, we discovered MA$_8$-1-hexanol is a special case, since it accommodates an aliphatic molecule: Hasegawa, T.; Iwamoto, T. *J. Inclusion Phenom.* **1988**, *5*, 549.

(17) Fernelius, W. C.; Burbage, J. J. *Inorg. Synth.* **1946**, *2*, 227.

(18) Konnert, K.; Hendrickson, W. A. *Acta Crystallogr., Sect. A* **1980**, *36*, 344.

Table II. Expected and Observed Occupanices in $P2/m$ of the Toluene Guest ($Z = 1$) for Possible Values of P_A[a]

atom	P_A				obsd
	1	0.67	0.5	0.33	
C(11)	0.5	0.5	0.5	0.5	0.498
C(12)	0.25	0.335	0.375	0.415	0.417
C(13)	0.25	0.25	0.25	0.25	0.251
C(14)	0.5	0.415	0.375	0.335	0.368
C(15)	0.25	0.25	0.25	0.25	0.309
tot.	1.75	1.75	1.75	1.75	1.843

[a] P_A is the fraction of the guest toluene molecules that are in orientation A (see text and Figure 1).

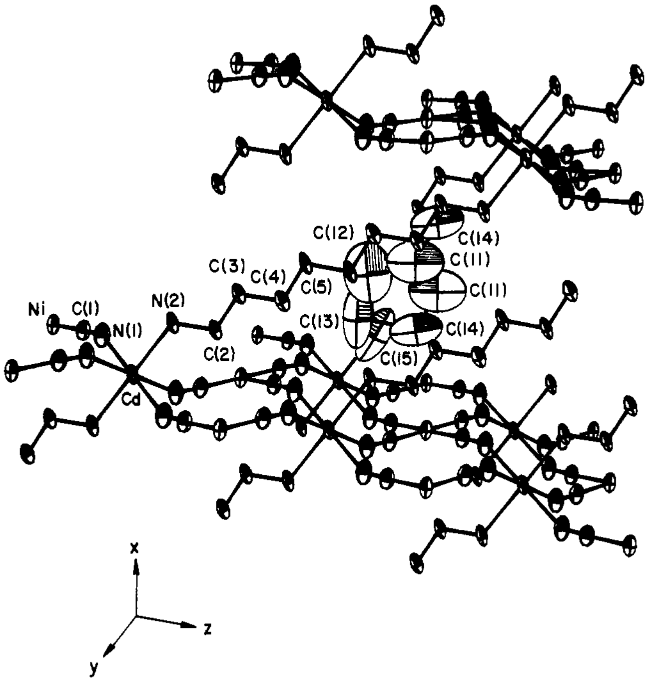

Figure 2. Side-view ORTEP drawing of MA_8–toluene. Only one complete α,ω-diaminooctane chain is shown for clarity. A toluene molecule is shown in only one of several possible orientations (see Discussion).

the refined orientations. In addition to higher R values, the other trial orientations all exhibited a distinct absence of electron density in the vicinity of the methyl group as reflected in the difference Fourier maps.

Parameters obtained from LSRE refinement were then used in a standard full-matrix least-squares calculation (LSFM) without restraints. This resulted in the merging of the four nearly overlapping atoms of the toluene guest. On the basis of this observed overlap, it is apparent from simple packing considerations that an observable population of the previously neglected orientation illustrated in Figure 1B might also be expected. In order to test this hypothesis, the occupancies of the toluene atoms were refined individually. Prior to this, the four overlapping atoms having occupancies of 0.25 were replaced with an equivalent model in which two nonoverlapping atoms had occupancies of 0.5. This simplification avoids the poorly conditioned problem of refining occupancies for nearly overlapping atoms. The observed occupancies are presented in Table II. Setting the initial occupancies of all toluene atoms to 0.5 or 0.25 or assigning random values between 0.25 and 0.5 led to results that differed by as much as 10% in absolute magnitude. The relative magnitudes of the occupancies, however, were better conserved.

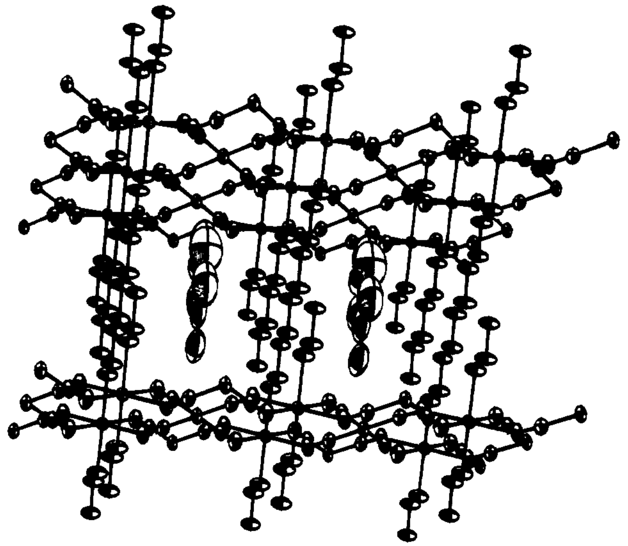

Figure 3. ORTEP drawing of MA_8–toluene, viewed through the channels of the host cage and showing the resident toluene guests. Toluene molecules are shown in only one of several possible orientations (see Discussion).

Table III. Fractional Atomic Coordinates and B_{eq} Values[a]

atom	x	y	z	B_{eq},[b] Å^2
Cd	0.0000	0.0000	0.0000	2.22 (1)
Ni	0.0000	0.5000	0.5000	2.47 (2)
N(1)	0.9417 (3)	0.2216 (5)	0.1883 (4)	3.73 (7)
N(2)	0.1959 (4)	0.0000	0.2106 (6)	4.10 (1)
C(1)	0.9613 (3)	0.3285 (5)	0.3057 (5)	2.88 (7)
C(2)	0.2112 (5)	0.0000	0.4273 (7)	4.1 (1)
C(3)	0.3430 (5)	0.0000	0.5484 (7)	4.0 (1)
C(4)	0.6412 (6)	0.0000	0.2313 (8)	4.4 (2)
C(5)	0.5065 (6)	0.0000	0.1127 (7)	4.9 (2)
C(11)	0.536 (2)	0.5000	0.440 (3)	20 (1)
C(12)	0.542 (4)	0.5000	0.813 (4)	25 (1)
C(13)	0.695 (5)	0.5000	0.869 (4)	23 (2)
C(14)	0.681 (2)	0.5000	0.556 (4)	15.9 (8)
C(15)	0.735 (3)	0.5000	0.776 (3)	20 (1)

[a] Parameters without standard deviations are fixed by symmetry. Occupancies for Cd and Ni are 0.25. Occupanices for C(11)–C(15) are given in Table II. [b] $B_{eq} = 8\pi^2/3\sum_i\sum_j U_{ij}$, where the general temperature factor expression is $\exp(-2\pi^2\{U_{11}h^2a^{*2} + U_{22}k^2b^{*2} + U_{33}l^2c^{*2} + 2U_{12}hka^*b^* + 2U_{13}hla^*c^* + 2U_{23}klb^*c^*\})$.

Table IV. Selected Bond Distances and Angles

Distances (Å)			
Cd–N(1)	2.357 (4)	C(5)–C(5)′	1.552 (7)
Cd–N(2)	2.306 (4)	C(11)–C(11)′	1.32 (3)
Ni–C(1)	1.859 (4)	C(11)–C(12)	1.76 (3)
N(1)–C(1)	1.140 (5)	C(11)–C(14)	1.63 (3)
N(2)–C(2)	1.487 (6)	C(12)–C(13)	1.67 (7)
C(2)–C(3)	1.503 (7)	C(13)–C(15)	0.90 (6)
C(3)–C(4)	1.511 (7)	C(14)–C(15)	1.50 (3)
C(4)–C(5)	1.525 (8)		
Angles (deg)			
N(1)–Cd–N(1)′	180.0	C(3)–C(4)–C(5)	112.2 (5)
N(1)–Cd–N(1)″	90.0 (1)	C(4)–C(5)–C(5)′	110.9 (5)
N(1)–Cd–N(2)	90.0 (1)	C(11)–C(11)′–C(12)	115 (2)
C(1)–Ni–C(1)′	180.0	C(11)–C(11)′–C(14)	113 (1)
C(1)–Ni–C(1)″	90.0 (2)	C(12)–C(11)′–C(14)	131 (2)
Cd–N(1)–C(1)	153.5 (3)	C(11)′–C(12)–C(13)	116 (2)
Cd–N(2)–C(3)	118.6 (4)	C(12)–C(13)–C(15)	122 (4)
Ni–C(1)–N(1)	177.7 (4)	C(11)–C(14)–C(15)	126 (2)
N(2)–C(2)–C(3)	113.4 (5)	C(13)–C(15)–C(14)	127 (4)
C(2)–C(3)–C(4)	113.5 (5)		

In the final stage of refinement, host hydrogen atoms with calculated positions were included for refinement and anisotropic thermal parameters were applied for all atoms except hydrogen. The final unweighted R value was 0.025. The largest positive peak in the difference Fourier map (0.449 e/Å^3) was located at (0.00,0.00,0.00), which is the position of the Cd atom. The largest negative peak in the difference map (0.421 e/Å^3) was also located near the Cd atom. There were twelve peaks more than three standard deviations above noise (estimated error in electron density = 0.88). Figure 2 shows an ORTEP[19] illustration of the structure along with the atomic notations; for the sake of clarity, not all atoms of the host cage are shown. The guest toluene molecules are shown in only one of several possible orientations relative to the host cage. Figure 3 presents a different ORTEP perspective of several adjacent unit cells, emphasizing the extended nature of the cage structure as well as the relative positions of the guest toluenes. Table III indicates the final

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(19) Johnson, C. K. ORTEP-II. Report ORNL-5138; Oak Ridge National Laboratory: Oak Ridge, TN, 1976.

atomic coordinates and thermal parameters. Table IV lists selected bond distances and angles.

Discussion

The structural analysis reveals this compound to be a typical MA. The host structure consists of cyanometal complex sheets, $[Cd(NC)_4Ni]$, extending in two dimensions (the floors), and bridging diaminooctanes (the pillars) linked by two Cd atoms. The nickel tetracyanide moiety is planar, and the bond angle formed by the cyanide and the Cd atom (C(1)–N(1)–Cd) is 153.5°. The Cd(1)–N(1), N(1)–C(1), and C(1)–Ni(1) distances were 2.357 (4), 1.140 (5), and 1.859 (4) Å, respectively, and are quite similar to the values obtained in other clathrate compounds of this type.[6] The 1,8-diaminooctane is in the all-trans conformation and coordinated to Cd at the amine N(2). The four cyanide nitrogens (N(1) and its C_2- and mirror-symmetry-related mates) and the two amine nitrogens (N(2) and its C_2-symmetry-related mate) give the Cd atom an octahedral site symmetry. The MA_8 host is presently the largest cage structure known in the MA series obtained by the method described in the Experimental Section. Another candidate for large cage structures, 1,9-diaminononane, has led to different structures.[20] Another interesting fact is that the center of the benzene ring in the toluene appears not to coincide with the center of symmetry of the unit cell, as is often the case in smaller clathrate structures.[10,13]

The large size of the cage relative to the toluene guest molecule gives rise to a greater degree of disorder than has been found in other clathrate structures in the series MA_n–xG. The approximate overlap in the positions of four atoms in the toluene guest with atoms of its C_2-symmetry-related mate lead to the prediction of a second disordered orientation, as depicted in Figure 1. We shall denote these two orientations by A and B and the corresponding fractional populations by P_A and P_B, respectively. A qualitative inspection of the density observed in the Fourier and difference Fourier maps suggests that a satisfactory description of the disorder would require $P_B > P_A$, since the electron density is greater at position 12 than position 14. This prediction is consistent with the values of the refined occupancies. It is evident from Figure 1 that the expected occupancies for positions 11, 13, and 15 should depend only on the overlap between atoms of the symmetry mates but not on the distribution of population between the two disordered orientations. The occupancies for positions 12 and 14, on the other hand, are expected to depend on both overlap as well the population of the disordered states. The predicted occupancies for several different values of P_A are given in Table II. While the observed occupancies appear to be modeled well by the case where $P_A = 0.33$, the error in the refined occupancies is too large to determine definitively how much one set of C_2-symmetry-related orientations contributes. Rather, the observed occupancies are consistent with some degree of disorder—specifically some distribution between these two sets of C_2-symmetry-related orientations that slightly favors P_B. Finally, we note that the thermal ellipsoids for the toluene atoms are quite large. Although some contribution to these derives from the thermally averaged motion of the toluene molecule within each unit cell, the unusual behavior of the thermal ellipsoids for the toluene guest is due in part to the errors inherent in our oversimplified model for the disordered toluene molecule. The observation of disorder in this structure is understandable given the large size of the cage relative to the toluene guest.

The large size of this cage has interesting possibilities for inclusion of larger guest molecules,[21,22] and we stress that the difficulties encountered in solving this crystal structure appear to be due to the unusual orientation of the toluene guest possible in the MA_8 clathrate structure. We do not rule out the possibility that the toluene guest is actually able to change its orientation at room temperature in a cage of this size. The energy barrier between the two orientations should not be large, and the shape of the thermal ellipsoids is consistent with such motions.

Acknowledgment. We are grateful to Dr. W. Dean Harman, presently at the University of Virginia, for assistance during the X-ray measurement, and to Dr. Toschitake Iwamoto, University of Tokyo, and Dr. Henry Taube, Stanford University, for reviewing the manuscript.

Supplementary Material Available: Tables listing equivalent atom labels, crystallographic details, thermal parameters, least-squares planes, and bond distances and angles for the atoms in the structure including hydrogen atoms (8 pages); a table of calculated and observed structure factors (5 pages). Ordering information is given on any current masthead page.

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(21) Iwamoto, T.; Nishikiori, S.; Hasegawa, T. *J. Inclusion Phenom.* **1987**, *5*, 225.

(22) Hasegawa, T. D.Sc. Thesis, University of Tokyo, 1986.