N-ethylsalicylaldiminato and nta^{3-} = nitrilotriacetato. However, sulfur and oxygen donors do not sort in this fashion.[21]

(17) Lewis, D. F.; Fay, R. C. *J. Chem. Soc., Chem. Commun.* **1974**, 1046.

(18) Bonds, W. D., Jr.; Archer, R. D.; Hamilton, W. C. *Inorg. Chem.* **1971**, *10*, 1764.

(19) Bradley, D. C.; Hursthouse, M. B.; Rendall, I. F. *J. Chem. Soc., Chem. Commun.* **1970**, 368.

(20) Hoard, J. L.; Willstadter, E.; Silverton, J. V. *J. Am. Chem. Soc.* **1965**, *87*, 1610.

(21) (a) Steffen, W. L.; Hawthorne, S. L.; Fay, R. C. *J. Am. Chem. Soc.* **1976**, *98*, 6757. (b) Hawthorne, S. L.; Bruder, A. H.; Fay, R. C. *Inorg. Chem.* **1978**, *17*, 2114. (c) Steffen, W. L.; Fay, R. C. *Ibid.* **1978**, *17*, 2120.

(22) The structural characterization of an analogous d^2 species [e.g., W(dsp)$_2$] would be particularly enlightening because the positions of the N and O donors in the dodecahedron should be reversed. Thus, the ligand would no longer span *m* and *a* edges. Instead, the reversal would cause an alternate wrapping such as *gbg*, which allows the ligands to wrap without severe distortions from planarity and with the N and O donors in the appropriate positions.[23] The two chelate ring *b* edges for this isomer would need to be somewhat shortened, which might lead to another isomer with less ligand planarity instead.

(23) Donahue, C. J. Ph.D. Dissertation, University of Massachusetts, Amherst, MA, 1977.

(24) (a) Bellamy, L. J.; Branch, R. F. *J. Chem. Soc.* **1954**, 4491. (b) Marvel, C. S.; Aspey, S. A.; Dudley, E. A. *J. Am. Chem. Soc.* **1956**, *78*, 4905. (c) Mehrotra, R. C.; Gupta, V. D.; Bharara, P. C. *Indian J. Chem.* **1973**, *11*, 814. (d) Biradar, N. S.; Goudar, T. R.; Mahale, V. B.; Kulkarni, V. H. *J. Karnatak Univ.* **1973**, *18*, 19.

(25) Percy, G. C.; Thornton, D. A., *J. Inorg. Nucl. Chem.* **1973**, *35*, 2719.

(26) Biradar, N. S.; Kulkarni, V. H., *Z. Anorg. Allg. Chem.* **1972**, *387*, 275.

(27) Bertrand, J. A.; Eller, P. G. *Inorg. Chem.* **1974**, *13*, 927.

(28) Tsuge, S.; Leary, J. J.; Isenhour, T. L. *Anal. Chem.* **1973**, *45*, 198.

(29) Silverton, J. V.; Hoard, J. L. *Inorg. Chem.* **1963**, *2*, 243.

Notes

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Synthesis and Structure of 1-Methyl-1,3,5,7-tetraazaadamantan-1-ium Octaiodide, $[(CH_2)_6N_4CH_3]_2I_8$. A New Outstretched *Z* Configuration for the Polyiodide Ion I_8^{2-}

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Among members of the halogen family, iodine exhibits the highest tendency for catenation, giving rise to a variety of polyiodide species of the general formula I_x^{n-}.[1] These anion complexes, which can be either discrete or polymeric, crystallize with large counterions such as Cs^+, R_4N^+, and protonated organic nitrogen compounds. Values of x ranging from 3 to 16 are known, with $n = 1$ for x odd and $n = 2$ or 4 for x even. While a rather large number of triiodides has been extensively investigated,[1,2] relatively little is known about the higher polyiodides, and the bulk of our knowledge about these systems is derived from X-ray crystallographic studies. The only well-established discrete higher polyiodide entities are I_4^{2-},[3,4] I_5^-,[5,6] I_8^{2-},[7] I_9^-,[8] and I_{16}^{4-}[9] (Table I). Other higher polyiodides, such as (phenacetinH)I_5,[10] (quinuclidineH)I_5,[11] $[(C_2H_5)_4N]I_7$,[12] and $[(\text{pyridine})_2I]I_7$,[13] adopt infinite network structures in which no separate polyiodide aggregates can be distinguished.

Recently we prepared[14] and determined the crystal and molecular structure[15] of hexamethylenetetramine oxide (1,3,5,7-tetraazaadamantane *N*-oxide), $(CH_2)_6N_4O$. In an attempt to prepare donor–acceptor complexes from the system $(CH_2)_6N_4O$–I_2–$CHCl_3$, we unexpectedly isolated the title compound as one of the products. Structural characterization of this new polyiodide, as reported in the present paper, revealed an outstretched form of the I_8^{2-} anion differing from the *Z* configuration observed in its cesium salt[7] (Figure 1).

Experimental Section

Hexamethylenetetramine oxide, $(CH_2)_6N_4O$, was prepared by the reaction of hexamethylenetetramine with 30% aqueous hydrogen peroxide and recrystallized from chloroform.[14] The title compound was obtained by slow evaporation of a chloroform solution of $(CH_2)_6N_4O$ (1.95×10^{-3} M) and I_2 (3.53×10^{-3}–5.30×10^{-3} M) in air at ambient temperatures. A small crop of black crystals with dark metallic luster was deposited after several days. When carbon tetrachloride was used to wash the crystals, a reddish violet color appeared in the resultant liquid. After the product had been stored in a clear plastic vial for a few hours, reddish violet stains appeared on the inside wall. These observations indicate that some I_2 molecules adhere loosely to the surface of freshly prepared crystals. Anal. Calcd for $[(CH_2)_6N_4CH_3]_2I_8$: C, 12.68; H, 2.28; N, 8.45. Found: C, 12.81; H, 2.22; N, 8.55. Upon heating, the black crystals changed irreversibly into a light orange color at about 127 °C and eventually melted at 160 °C. A better yield of the compound was obtained by reacting equivalent quantities of $(CH_2)_6N_4$ and CH_3I and an excess of I_2 in chloroform. However, two other kinds of relatively unstable orange and reddish brown crystals appeared in the same preparation. These side products were suspected to be $(CH_2)_6N_4{\cdot}I_2$[16] and $(CH_2)_6N_4{\cdot}2I_2$[16] and were manually separated from the black crystals of $[(CH_2)_6N_4CH_3]_2I_8$.

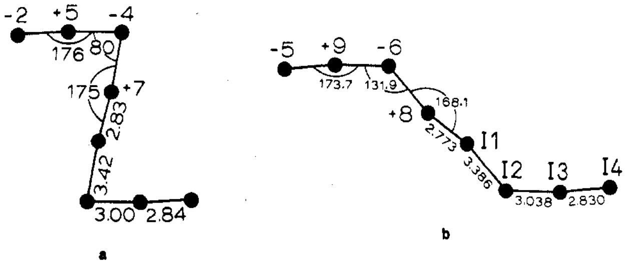

Figure 1. Dimensions of the I_8^{2-} ion in (a) Cs_2I_8 and (b) $[(CH_2)_6N_4CH_3]_2I_8$. The signed figures represent deviations (in Å × 10^{-2}) of atoms from an exactly planar configuration.

A crystal in the shape of a polyhedron with cross sections of 0.2–0.45 mm was selected. Preliminary precession photographs and accurate measurements on a Nonius CAD-4 diffractometer with graphite monochromator and Mo Kα radiation (λ 0.71069 Å) indicated space group $P2_1/c$ and the following cell parameters: $a = 11.258$ (2) Å, $b = 9.827$ (1) Å, $c = 14.150$ (3) Å, $\beta = 92.08$ (1)°, and $V = 1564.4$ Å^3. For $Z = 2$ and a molecular weight of 1325.74, the calculated density is 2.814 g cm^{-3}, in reasonable agreement with the measured density of 2.79 (2) g cm^{-3} by flotation in carbon tetrachloride/1,1,2,2-tetrabromoethane.

Intensities for 4540 unique reflections with $\theta < 30°$ were collected by using the ω-scan technique, and three standard reflections were measured every hour to check the stability of the crystal and the electronics. Data were corrected for absorption ($\mu = 80.4$ cm^{-1}) by the Gaussian integration method,[17,18] and transmission factors ranged from 0.031 to 0.151. Of the complete data set, 2736 independent reflections (60.3%) had intensities greater than $3\sigma(I)$ above background.

The structure was solved by direct methods, and all 15 nonhydrogen atoms in the asymmetric unit were refined anisotropically by full-matrix least-squares techniques. In the last cycle all 12 methylene H atoms were introduced and assigned the same isotropic temperature factor $B = 5.0$ Å^2, yielding a conventional discrepancy index $R \equiv \sum||F_o| - |F_c|| / \sum|F_o| = 0.096$. The final difference map did not reveal

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Table I. Intramolecular Distances and Shortest Intermolecular Contacts in Some Discrete Higher Polyiodides[a]

	polyhalide ion	shape	I–I, Å	I···I, Å	ref
I_2, 20 °C			2.68	3.56	22
I_2, −163 °C			2.715	3.50	23
$[Cu(NH_3)_4]I_4$	I_4^{2-}	linear	2.80, 3.34 (2×)	4.65	3
Tl_6PbI_6	I_4^{2-}	linear	3.14, 3.18 (2×)	4.03	4[b]
$[(CH_3)_4N]I_5$	I_5^-	*V*	2.82 (2×), 3.17 (2×)	3.63	5
$C_{40}H_{36}FeN_6O_4I_5$[c]	I_5^-	*V*	2.77, 2.81, 3.04, 3.13	4.02	6
Cs_2I_8	I_8^{2-}	*Z*	2.83, 2.84 (2×), 3.00 (2×), 3.42 (2×)	3.90	7
$[(CH_2)_6N_4CH_3]_2I_8$	I_8^{2-}	outstretched *Z*	2.77, 2.83 (2×), 3.04 (2×), 3.39 (2×)	4.28	*d*
$[(CH_3)_4N]I_9$	I_9^-	twisted *h*	2.67 (2×), 2.90, 2.91, 3.18, 3.24 (2×), 3.43	3.49	8
$(theobromine)_2 \cdot H_2I_8$	I_{16}^{4-}	*S*	2.76 (2×), 2.84 (2×), 2.92 (2×), 2.94 (2×), 3.03 (2×), 3.35 (2×), 3.42 (2×), 3.45	3.84	9

[a] Relevant distances in crystalline iodine[22,23] are included for the sake of comparison. [b] The distances calculated from the given cell dimensions and atomic coordinates differ slightly from those quoted in the text. [c] Bis(α-benzyldioximato)bis(β-picoline)iron(III) pentaiodide; IUPAC name: bis[(diphenylethanedione dioximato)(1−)-*N*,*N*′]bis(3-methylpyridine)iron(1+) pentaiodide(1−). [d] This work.

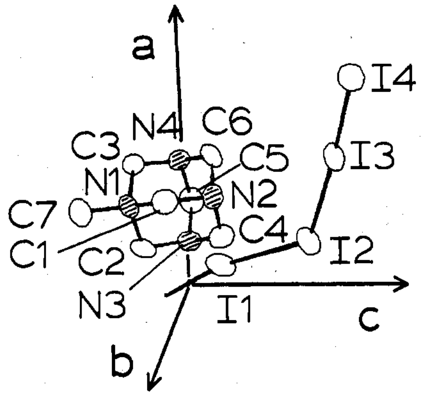

Figure 2. Atom numbering of the asymmetric unit in $[(CH_2)_6N_4CH_3]_2I_8$.

plausible positions for the methyl H atoms. The function minimized was $\sum w(|F_o| - |F_c|)^2$, with $w = 1/\sigma^2(F_o)$. Scattering factors for neutral atoms[19] and spherically bonded hydrogen[20a] were generated from the coefficients of analytical approximations; the real and imaginary parts of the anomalous dispersion correction were included only for neutral I.[20b] The atom numbering of the asymmetric unit is shown in Figure 2, and the final positional parameters appear in Table II. Dimensions of the $[(CH_2)_6N_4CH_3]^+$ cation are listed in Table III. Thermal parameters (Table IV), positions of generated methylene H atoms (Table V), comparison of observed and calculated structure factors (Table VI), and a stereoview of the molecular packing (Figure 3) are available as supplementary material.

Results and Discussion

The present analysis has established that the title compound is a 2:1 salt of the 1-methyl-1,3,5,7-tetraazaadamantan-1-ium cation and the octaiodide anion. It is not clear how the methyl group, presumably originating from a solvent chloroform molecule, displaces the *N*-oxide function in the heterocyclic cage system.

All higher polyiodides studied to date consist of molecular aggregates of I_2, I^-, and I_3^-, the interaction among which ranges from rather strong to fairly weak. The I_8^{2-} as found in Cs_2I_8[7] and in the present structure can be considered to be a central I_2 molecule with two asymmetric, slightly bent I_3^- arms pointing in opposite directions (Figure 1). Both configurations are essentially planar and each possesses a center of symmetry. As compared to the well-known *Z* configuration (Figure 1a) for the I_8^{2-} ion in the Cs^+ salt, the present one differs mainly in the apex angle at I(2) (131.9° vs. 80° in Cs_2I_8), resulting in an extended structure with a flatter profile and longer reach (Figure 1b). The dimensions of the I_3^- moiety in both configurations are virtually identical. However, the central portion of the present I_8^{2-} ion has developed a pronounced kink in the reverse sense and is also more strongly bonded together.

Table II. Final Positional Parameters[a] for $[(CH_2)_6N_4CH_3]_2I_8$

atom	*x*	*y*	*z*
I(1)	0.0550 (1)	0.0706 (1)	0.0743 (1)
I(2)	0.1308 (1)	0.2625 (1)	0.2659 (1)
I(3)	0.3611 (1)	0.4043 (1)	0.3262 (1)
I(4)	0.5700 (1)	0.5571 (1)	0.3662 (1)
N(1)	0.247 (1)	0.645 (1)	−0.097 (1)
N(2)	0.272 (1)	0.692 (2)	0.075 (1)
N(3)	0.150 (1)	0.504 (1)	0.018 (1)
N(4)	0.360 (1)	0.490 (1)	0.006 (1)
C(1)	0.263 (1)	0.755 (1)	−0.020 (1)
C(2)	0.134 (1)	0.563 (2)	−0.075 (1)
C(3)	0.352 (1)	0.549 (2)	−0.086 (1)
C(4)	0.166 (1)	0.610 (2)	0.087 (1)
C(5)	0.252 (2)	0.414 (1)	0.025 (1)
C(6)	0.374 (1)	0.599 (2)	0.075 (1)
C(7)	0.236 (2)	0.705 (2)	−0.192 (1)

[a] In this and the following table the numbers in parentheses are the estimated standard deviations for the least significant figures.

Table III. Bond Lengths (Å) and Angles (deg) in the $[(CH_2)_6N_4CH_3]^+$ Cation

Bond Lengths			
N(1)–C(7)	1.47 (2)		
N(1)–C(1)	1.53 (2)	N(2)–C(4)	1.46 (2)
N(1)–C(2)	1.55 (2)	N(2)–C(6)	1.47 (2)
N(1)–C(3)	1.51 (2)	N(3)–C(4)	1.43 (2)
N(2)–C(1)	1.48 (2)	N(3)–C(5)	1.45 (2)
N(3)–C(2)	1.45 (2)	N(4)–C(5)	1.46 (2)
N(4)–C(3)	1.43 (2)	N(4)–C(6)	1.45 (2)
Angles around Quaternary N Atom (Mean CH_3–N–C = 111°, C–N–C = 107°)			
C(7)–N(1)–C(1)	112 (1)	C(1)–N(1)–C(2)	108 (1)
C(7)–N(1)–C(2)	110 (1)	C(1)–N(1)–C(3)	107 (1)
C(7)–N(1)–C(3)	112 (1)	C(2)–N(1)–C(3)	107 (1)
Angles around Tertiary N Atoms (Mean C–N–C = 109°)			
C(1)–N(2)–C(4)	108 (1)	C(4)–N(3)–C(5)	109 (1)
C(1)–N(2)–C(6)	107 (1)	C(3)–N(4)–C(5)	110 (1)
C(4)–N(2)–C(6)	107 (1)	C(3)–N(4)–C(6)	108 (1)
C(2)–N(3)–C(4)	110 (1)	C(5)–N(4)–C(6)	110 (1)
C(2)–N(3)–C(5)	112 (1)		
Angles around Methylene C Atoms (Mean N–C–N = 111°)			
N(1)–C(1)–N(2)	111 (1)	N(2)–C(4)–N(3)	114 (1)
N(1)–C(2)–N(3)	108 (1)	N(3)–C(5)–N(4)	110 (1)
N(1)–C(3)–N(4)	111 (1)	N(2)–C(6)–N(4)	113 (1)

To our knowledge the structure of the $[(CH_2)_6N_4CH_3]^+$ cation (Table III) has been determined for the first time. Of primary interest are the unequal C–N bonds involving the formally positive quaternary N(1) atom. The N(1)–CH_2 bond [mean = 1.53 (2) Å] is significantly longer than the exocyclic N(1)–CH_3 bond [1.47 (2) Å] and other C–N bonds in the heterocyclic ring. The remaining dimensions of the cation are in good agreement with those in $(CH_2)_6N_4$[21] and in $(CH_2)_6N_4O$.[15]

As a consequence of the large cationic size in the present structure, the I_8^{2-} aggregates are well separated from one another. The observed nonbonded I···I contact of 4.28 Å is the second largest among discrete higher polyiodides and is appreciably longer than the corresponding interionic distance in Cs_2I_8 (Table I).

Registry No. $[(CH_2)_6N_4CH_3]_2I_8$, 70955-00-9; $(CH_2)_6N_4O$, 62190-92-5; $(CH_2)_6N_4$, 100-97-0; CH_3I, 74-88-4; I_2, 7553-56-2.

Supplementary Material Available: Thermal parameters (Table IV), positions of generated methylene H atoms (Table V), comparison of observed and calculated structure factors (Table VI), and a stereoview of the molecular packing (Figure 3) (43 pages). Ordering information is given on any current masthead page.

References and Notes

(1) (a) E. H. Wiebenga, E. E. Havinga, and K. H. Boswijk, *Adv. Inorg. Chem. Radiochem.*, **3**, 133–69 (1961); (b) A. I. Popov in "Halogen Chemistry", Vol. 1, V. Gutmann, Ed., Academic Press, London, 1967, pp 225–264; (c) *MTP Int. Rev. Sci.: Inorg. Chem., Ser. One*, **3**, 53–84 (1972); (d) A. I. Popov and T. Surles, *MTP Int. Rev. Sci.: Inorg. Chem., Ser. Two*, **3**, 179–83 (1975); (e) A. J. Downs and C. J. Adams in "Comprehensive Inorganic Chemistry", Vol. 2, J. C. Bailar, Jr., H. J. Emeléus, R. Nyholm, and A. F. Trotman-Dickinson, Eds., Pergamon, Oxford, 1973, pp 1534–63; (f) F. K. Tebbe, *Acta Crystallogr., Sect. A*, **34**, S149 (1978).

(2) J. Runsink, S. Swen-Walstra, and T. Migchelsen, *Acta Crystallogr., Sect. B*, **28**, 1331 (1972), and references therein.

(3) E. Dubler and L. Linowsky, *Helv. Chim. Acta*, **58**, 2604 (1975).

(4) A. Rabenau, H. Schulz, and W. Stoeger, *Naturwissenschaften*, **63**, 245 (1976).

(5) J. Broekema, E. E. Havinga, and E. H. Wiebenga, *Acta Crystallogr.*, **10**, 596 (1957).

(6) A. A. Dvorkin, Yu. A. Simonov, T. I. Malinovskii, I. I. Bulgak, and D. G. Batyr, *Dokl. Akad. Nauk SSSR*, **234**, 1372 (1977).

(7) E. E. Havinga, K. H. Boswijk, and E. H. Wiebenga, *Acta Crystallogr.*, **7**, 487 (1954).

(8) W. J. James, R. J. Hach, D. French, and R. E. Rundle, *Acta Crystallogr.*, **8**, 814 (1955).

(9) F. H. Herbstein and M. Kapon, *J. Chem. Soc., Chem. Commun.*, 677 (1975).

(10) F. H. Herbstein and M. Kapon, *Nature (London), Phys. Sci.*, **239**, 153 (1972).

(11) J. Jander, H. Pritzkow, and K.-U. Trommsdroff, *Z. Naturforsch., B*, **30**, 720 (1975).

(12) E. E. Havinga and E. H. Wiebenga, *Acta Crystallogr.*, **11**, 733 (1958).

(13) O. Hassel and H. Hope, *Acta Chem. Scand.*, **15**, 407 (1961).

(14) Y.-S. Lam and T. C. W. Mak, *Aust. J. Chem.*, **31**, 1249 (1978).

(15) T. C. W. Mak, M. F. C. Ladd, and D. C. Povey, *J. Chem. Soc., Perkin Trans. 2*, in press.

(16) H. Pritzkow, *Acta Crystallogr., Sect. B*, **31**, 1589 (1975).

(17) P. Coppens, L. Leiserowitz, and D. Rabinovich, *Acta Crystallogr.*, **18**, 1035 (1965).

(18) W. R. Busing and H. A. Levy, *Acta Crystallogr.*, **22**, 457 (1967).

(19) D. T. Cromer and J. B. Mann, *Acta Crystallogr., Sect. A*, **24**, 321 (1968).

(20) "International Tables for X-ray Crystallography", Vol. IV, Kynoch Press, Birmingham, England, 1974: (a) pp 99–101; (b) pp 149–50.

(21) L. N. Becka and D. W. J. Cruickshank, *Proc. R. Soc. London, Sect. A*, **273**, 435 (1963).

(22) A. I. Kitaigorodskii, T. L. Khotsyanova, and Yu. T. Struchkov, *Zh. Fiz. Khim.*, **27**, 780 (1953).

(23) F. van Bolhuis, P. B. Koster, and T. Migchelsen, *Acta Crystallogr.*, **23**, 90 (1967).

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Dimeric (Pentamethylcyclopentadienyl)rhodium and -iridium Complexes. 6.[1] Crystal Structure of $[(\eta^5\text{-}C_5Me_5)RhI]_2(\mu\text{-}I)_2\cdot 2C_6H_5Me$ and Systematics in the Series $[(\eta^5\text{-}C_5Me_5)RhX]_2$ (X = Cl, Br, I)

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In conjunction with studies on molecules containing bridging hydride ligands, e.g., $[(\eta^5\text{-}C_5Me_5)RhCl]_2(\mu\text{-}H)(\mu\text{-}Cl)$,[2] we have undertaken single-crystal X-ray diffraction studies on the parent rhodium(III) dihalides, $[(\eta^5\text{-}C_5Me_5)RhX]_2(\mu\text{-}X)_2$ (X = Cl, Br, I). Details concerning the chloride[3] and bromide[4] species have appeared previously; for completeness, we now report the results of a structural study on the final member of this series, $[(\eta^5\text{-}C_5Me_5)RhI]_2(\mu\text{-}I)_2$.

Table I. Crystal Data for $[(\eta^5\text{-}C_5Me_5)RhI]_2(\mu\text{-}I)_2\cdot 2C_6H_5Me$

A. Cell Parameters at 24 °C[a]

crystal system: monoclinic	$a = 13.9414\ (18)$ Å
space group: $C2/m\ [C_{2h}^3$; No. 12]	$b = 11.2421\ (14)$ Å
$Z = 2$	$c = 11.9675\ (15)$ Å
mol wt 1168.18	$\beta = 90.406\ (10)°$
ρ(calcd) = 2.068 g cm^{-3}	$V = 1875.6\ (4)$ Å^3

B. Collection of Intensity Data

diffractometer: Syntex $P2_1$
radiation: Mo Kα (λ 0.710 73 Å)
monochromator: pyrolitic graphite; equatorial
reflctns measd: $+h,\pm k,\pm l$ (two forms)
2θ range: 4.0–45.0°
scan type: coupled θ(crystal)–2θ(counter)
scan speed: 3.0 °/min in 2θ
scan range: $[2\theta(K\alpha_1) - 1.0]°$ to $[2\theta(K\alpha_2) + 1.0]°$
bkgd: stationary crystal and counter at beginning and end of 2θ scan; each for half of total scan time
standards: 3 every 97 reflections; no measurable decay
reflctns collected: 2589 total, yielding 1311 independent
data averaging: $R(I) = 2.27\%$ (after correction for absorption) for 1174 data having two or more contributions
absn coeff: $\mu = 41.3$ cm^{-1}
absn correction: empirical, based on ψ scans of 8 reflections[b]

[a] Based on 24 reflections with $27° < 2\theta < 29°$, well dispersed in reciprocal space. [b] Reflections used, their 2θ values, and their maximum:minimum transmission ratios were as follows: $3\bar{5}1$ and $\bar{3}5\bar{1}$, 20.5°, 1.12:1; 371 and $\bar{3}7\bar{1}$, 27.3°, 1.15:1; $5\bar{9}1$ and $\bar{5}9\bar{1}$, 36.4°, 1.14:1; $6,\bar{10},1$ and $\bar{6},10,\bar{1}$, 41.2°, 1.14:1.

Experimental Section

The complex was prepared by metathesis of $[(\eta^5\text{-}C_5Me_5)RhCl]_2(\mu\text{-}Cl)_2$ with sodium iodide in acetone. Attempts to obtain crystals from acetone solution yielded mainly thin plates; the material was finally recrystallized from toluene. A crystal of approximate dimensions 0.3 × 0.2 × 0.15 mm was mounted along its extended dimension (b) and transferred to our Syntex $P2_1$ diffractometer. Crystal alignment, determination of cell parameters, and data collection were carried out as described previously.[5] Details are given in Table I.

All computations were performed by using our Syntex XTL system.[6] The structure was solved via the multiple tangent formula method, using MULTAN,[7] which revealed the $Rh_2I_2(\mu\text{-}I)_2$ core of the molecule. A difference Fourier synthesis revealed all nonhydrogen atoms (including a toluene of crystallization). Refinement and a further difference Fourier synthesis led to the location of all hydrogen atoms in the structure. Continued full-matrix least-squares refinement of positional parameters for all atoms, anisotropic thermal parameters for nonhydrogen atoms, and isotropic thermal parameters for the hydrogen atoms led smoothly to convergence with $R_F = 2.0\%$, $R_{wF} = 2.2\%$, and GOF = 1.074 for all 1311 independent reflections ($R_F = 1.8\%$ and $R_{wF} = 2.1\%$ for those 1221 reflections with $|F_o| > 3\sigma(F_o)$). The secondary extinction parameter, k, was 3.327×10^{-5}; this modifies $|F_o|$ as shown in eq 1. The greatest feature on a final difference Fourier

$$|F_o|^{\mathrm{cor}} = |F_o|^{\mathrm{uncor}}(1.0 + kI_o) \qquad (1)$$

synthesis was of height 0.46 e Å^{-3} and was close to a heavy-atom position; the structure is thus complete. Final positional and thermal parameters are collected in Table II.

Discussion

The $[(\eta^5\text{-}C_5Me_5)RhI]_2(\mu\text{-}I)_2$ molecule is centered on the position ($^1/_2$, 0, 0) and has crystallographically imposed C_{2h} ($2/m$) symmetry. Atoms I(T), Rh, C(1), C(4), and H(4B)

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