precedent for the coupling of a triphenylphosphine ligand to an η-cyclopentadienyl group. The mechanism of this coupling and its role in the overall reaction sequence are not clear.

Acknowledgment. This work was supported in part by the National Science Foundation.

Registry No. $\overline{RuC_6F_4N{=}N}C_6F_5(Ph_2PC_6H_4\text{-}\eta\text{-}C_5H_4)$, 59569-15-2.

Supplementary Material Available: A listing of structure factor amplitudes and Table VI, giving principal amplitudes of thermal motion (23 pages). Ordering information is given on any current masthead page.

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Unusual Hydrogen Bonds. A Neutron Diffraction Study of the Hydrogen Dinitrate Ion, $(O_2NO{\cdot}H{\cdot}ONO_2)^-$, in Cesium Hydrogen Dinitrate[1]

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Received April 7, 1976 AIC60260Z

The crystal structure of cesium hydrogen dinitrate, $Cs^+(O_2NO{\cdot}H{\cdot}ONO_2)^-$, has been determined from three-dimensional single-crystal neutron-diffraction data. The crystals are orthorhombic, space group *Fddd*, with eight formula units in the unit cell of dimensions $a = 12.527$ (6) Å, $b = 7.344$ (3) Å, and $c = 12.983$ (6) Å. A full-matrix least-squares refinement based on F_o^2 gave a final R value of 0.050 for 391 independent reflections (derived from 1059 measured data) with intensities greater than $\sigma(F_o^2)$. The hydrogen dinitrate ion consists of two nitrate groups related by a very short [O⋯O = 2.468 (8) Å] and symmetric hydrogen bond. In the crystal the hydrogen dinitrate ions are shown to be orientationally disordered. This orientational disorder was not recognized in the previous x-ray studies and resulted in an apparent pseudo-tetrahedral coordination of the hydrogen atom and (O⋯O) ≃ 2.8–3.1 Å. Analysis of the thermal motion of the bridging hydrogen atom is consistent with a symmetric single-minimum potential well in the short hydrogen bond.

Introduction

As has been discussed in a previous x-ray structure publication,[2] the hydrogen dinitrate ion, $(O_2NO{\cdot}H{\cdot}ONO_2)^-$, apparently occurs in the crystalline state in two distinctly different configurations.[3,4] The first configuration, observed only in the tetraphenylarsonium (TPA) salt,[3] consists of two coplanar unidendate nitrate groups linked across a center of symmetry by a very short hydrogen bond (O⋯O ≃ 2.45 Å) and is very similar to that of some acid salts of organic monobasic acids referred to as type A using Speakman's classification.[5] The second and most frequently occurring configuration involves four oxygen atoms of two nitrate groups which surround a bridging hydrogen atom forming a distorted tetrahedron. The latter, rather unusual, configuration has been observed with cations of various sizes such as $[Rh(py)_4Cl_2]^+$ [4] and $(CH_3)_4N^+$.[6] In all cases at least one twofold symmetry axis relates the two nitrate groups. Although the $H(NO_3)_2^-$ infrared spectrum has been previously interpreted[7] in terms of a short (at most 2.5 Å O⋯O separation) and quasi-symmetrical hydrogen bond, the O⋯O distances observed in the x-ray studies appear to be much longer and range between 2.85 and 3.15 Å. The stability of the tetrahedral configuration has been shown recently to have a sound theoretical basis.[8] This neutron diffraction study of $CsH(NO_3)_2$ was first undertaken to obtain precise information on the location and dynamics of the bridging hydrogen atom in a simple system with a tetrahedrally coordinated hydrogen. While the results (vide infra) were somewhat unexpected they completely reconcile

Table I. Positional and Thermal Parameters for $CsNO_3 \cdot HNO_3$ and Root-Mean-Square Thermal Displacements (in Å) of Atoms along Their Principal Ellipsoid Axes[a,b]

Atoms	x	y	z	β_{11}	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}	μ_1	μ_2	μ_3
Cs	3750	3750	3750	43 (1)	173 (5)	56 (1)	0	0	0	184 (2)	218 (3)	219 (3)
O(1)	3711 (2)	1538 (17)	1289 (14)	39 (1)	332 (27)	114 (3)	−4 (3)	14 (3)	49 (10)	169 (3)	269 (9)	342 (11)
O(2)	2171 (4)	1845 (9)	1994 (5)	39 (2)	304 (14)	66 (2)	−11 (3)	6 (1)	50 (5)	175 (4)	206 (4)	313 (7)
O(3)	2389 (5)	91 (9)	683 (5)	90 (4)	290 (14)	69 (3)	−7 (5)	12 (3)	61 (5)	191 (4)	265 (6)	320 (7)
N	2782 (1)	1049 (15)	1283 (12)	43 (1)	147 (16)	56 (2)	−2 (1)	6 (1)	2 (5)	179 (2)	200 (11)	221 (4)
H	1250	1250	1933 (4)	87 (4)	275 (13)	61 (3)	10 (6)	0	0	228 (6)	258 (6)	279 (7)

[a] The estimated standard deviations in parentheses in this and all subsequent tables refer to the least significant figure. Positional parameters are multiplied by 10^4. [b] The anisotropic temperature factors are of the form: $\exp[-(\beta_{11}h^2 + \beta_{22}k^2 + \beta_{33}l^2 + 2\beta_{12}hk + 2\beta_{13}hl + 2\beta_{23}kl)]$. Their values are multiplied by 10^4 and the rms displacement values are multiplied by 10^3.

the structure with the spectroscopic data indicating the existence of a very strong hydrogen bond.[2,9]

Experimental Section

Crystal Preparation. Cesium hydrogen dinitrate was first described by Schultz[10] and the material used in this study was prepared as described by Wells and Metzger.[11] The observed melting point was in agreement with their reported value of 100 °C. More recently an incongruent melting point and a transition temperature of 104 °C have been reported.[12]

Crystals of suitable size for neutron diffraction study were grown by slow evaporation at room temperature of a saturated nitric acid solution. The crystal selected was bipyramidally shaped, exhibiting {1 2 1} boundary planes, and weighed 69.8 mg. The crystal was coated with Kel-F grease to prevent HNO_3 loss and sealed in a thin-walled lead-glass tube for data collection.

Crystal Data. The neutron study confirms the orthorhombic space group *Fddd* and the unit cell constants previously determined in the x-ray[2] study. Precise unit cell constants were determined using 30 intense reflections ranging in 2θ from 40 to 60°. A least-squares fit of the angles 2θ, χ, and ϕ of these reflections measured at 22 ± 2 °C with a neutron wavelength of 1.142 (1) Å yielded $a = 12.527$ (6) Å, $b = 7.344$ (3) Å, and $c = 12.983$ (6) Å. The density calculated assuming eight formula units per unit cell is 2.87 g cm^{-3} which agrees reasonably well with the value determined experimentally[2] of 2.73 g cm^{-3}.

Data Collection. Three-dimensional neutron data were collected using an Electronics and Alloys four-circle diffractometer at the CP-5 reactor at Argonne National Laboratory with a neutron wavelength of 1.142 (1) Å and using the θ–2θ step-scan technique. Step intervals of 0.1°, and preset scan ranges of 55–75 steps, were used with background intensity measurements obtained on both sides of each peak. The fully automated diffractometer operates under remote Sigma 5 computer control.

As a check of the crystal, and in order to monitor instrument stability, two reference reflections were measured every 30 reflections and were found to have a maximum random variation of 4%. Numerous reflections were purposely duplicated as a check during data collection and were found to be of constant intensity. For the complete data set, 1059 reflections were measured at 22 ± 2 °C from two octants of the reciprocal lattice out to $2\theta = 110°$. The observed integrated intensity of each reflection was corrected for absorption ($\mu_{calcd} \simeq 0.69$ cm^{-1}) and the corresponding transmission factors ranged from 0.77 to 0.91. All the equivalent data (symmetry equivalent and duplicate reflections) were averaged to yield a total of 465 independent reflections of which 391 had $(F_o^2) > 1.0\sigma(F_o^2)$. The "agreement factor" R, based on F_o^2, between averaged reflections was 0.048 where $R = \Sigma|(F_o^2)_{av} - F_o^2|/\Sigma F_o^2$. All the data were placed on an absolute scale by calibration with a well characterized NaCl crystal.[13]

Structure Refinements.[14] Inspection of preliminary Fourier and difference Fourier maps, phased using the previously determined x-ray atom positional parameters,[2] revealed two concentrations of negative scattering density corresponding to 0.5 proton $Å^{-3}$, separated by ~1.8 Å, and each near the midpoint along the line relating two symmetry equivalent oxygen atoms of different nitrate groups. Full-matrix least-squares refinement was initiated by varying a scale factor (S) and atom positional parameters (with isotropic temperature factors) and assuming the special position ($^1/_8$, $^1/_8$, z) for each half-hydrogen atom. The function minimized was $\Sigma w_i|F_o^2 - F_c^2|$ with $w = 1/\sigma(F_o^2)$ and $\sigma(F_o^2) = \sigma_c(F_o^2) + 0.02F_o^2$ where σ_c is determined by counting statistics. The agreement factors defined by the expressions $R(F_o) = \Sigma||F_o| - |F_c||/\Sigma|F_o|$, $R(F_o^2) = \Sigma|F_o^2 - F_c^2|/\Sigma F_o^2$, and $wR(F_o^2) = [\Sigma w_i(F_o^2 - F_c^2)^2/\Sigma w_i F_o^4]^{1/2}$ were $R(F_o) = 0.24$, $R(F_o^2) = 0.31$, and $wR(F_o^2) = 0.42$ for 391 reflections with intensities greater than $1.0\sigma(F_o^2)$. After six cycles of least-squares refinement, including anisotropic thermal parameters, the convergence appeared complete with $R(F_o) = 0.083$, $R(F_o^2) = 0.093$, and $wR(F_o^2) = 0.120$. At this stage of refinement the atomic positions and thermal parameters of the nonhydrogen atoms were in good agreement with those derived in the x-ray study and examination of a final difference Fourier map did not reveal any residual scattering density higher than 2.5% of a nitrogen atom. However, although the agreement factors were not unreasonably high, the value of Σ_1, the standard deviation of an observation of unit weight (defined by the relation $\Sigma_1 = [w_i|F_o^2 - F_c^2|/(n - p)]^{1/2}$ where n is the number of observations and p the number of parameters varied), remained relatively high at 3.53. This suggested that some inadequacy existed in the model used. Furthermore the double peak representing two half-hydrogen atoms raised the question of the nature of the disorder. It was not immediately discernible from the difference Fourier maps whether or not the disorder involved only the hydrogen atom bridging the nitrate groups or if a more complicated disorder existed which involved superposition of two hydrogen dinitrate groups. However, in the difference Fourier maps, the residual scattering density along the z axis, between the two half-hydrogen atoms, was at background level and seemed to preclude any possibility of a dynamic equilibrium between the two hydrogen positions (on the neutron diffraction time scale). It was at this point in the refinement that it was decided to attempt refinement using disordered hydrogen dinitrate groups because of the unexpected finding that two of the derived N–O distances in the hydrogen dinitrate ion were nearly *equal*, which might be an artifact due to disorder, especially since unequal N–O distances were expected because of the spectroscopic results indicating the formation of a strong hydrogen bond. Therefore a model was refined in which all atoms of the hydrogen dinitrate ion were disordered. Initial values for the oxygen atom positions, O(2) and O(3), were calculated using nonhydrogen bonded and hydrogen bonded N–O bond lengths of 1.20 and 1.30 Å previously reported.[2,3] In addition, small arbitrary shifts from the x axis were applied to the O(1) and N atomic positional parameters. The improvement in the refinements was quite dramatic and after a few cycles the convergence was complete with the final change to esd ratio being less than 0.1 for most parameters with a maximum ratio of 0.25. The final agreement factors were $R(F_o) = 0.052$, $R(F_o^2) = 0.050$, and $wR(F_o^2) = 0.063$ for all reflections with intensities greater than $1.0\sigma(F_o^2)$. The final scale factor was 0.766 (4), the isotropic extinction coefficient was 0.06 (2),[15] and the value of Σ_1 (standard deviation of an observation of unit weight) decreased from 3.53 to 1.92. As predicted the correlation coefficients were much higher than for the former ordered model and consequently the standard deviations in the refined parameters are about three times greater than those obtained for the ordered model. As explained in the Discussion section, the final refined model yielded O···O separations which were much shorter and which, chemically speaking, completely reconciled the spectroscopic and diffraction data. The former had indicated the presence of a strong hydrogen bond in the $H(NO_3)_2^-$ ion in $CsH(NO_3)_2$.[16]

The final positional and thermal parameters are given in Table I. In all the refinements the values assumed for the coherent neutron scattering amplitudes for Cs, O, N, and H were 0.54, 0.58, 0.94, and −0.374 (in units of 10^{-12} cm).[17]

Discussion

Successful refinement of the disordered $H(NO_3)_2^-$ group model appears to provide a meaningful description of the

Table II. Interatomic Distances and Angles around the Cesium Ion

Symmetry Operations Implied by the Subscripts Below

(a) $^5/_4 - x, ^1/_4 + y, ^1/_4 + z$
(b) $-^1/_4 + x, ^3/_4 - y, ^1/_4 + z$
(c) $x, ^1/_2 + y, ^1/_2 + z$
(d) $1 - x, ^1/_2 - y, ^1/_2 + z$
(e) $^1/_4 + x, ^1/_4 - y, ^1/_4 + z$
(f) $^3/_4 - x, ^3/_4 + y, ^1/_4 + z$
(g) $1 - x, 1 - y, z$
(h) $^1/_4 - x, ^1/_4 - y, z$

Interatomic Distances, Å

Cs· · ·O(1)$_a$	3.189 (2)	Cs· · ·O(3)$_e$	3.246 (7)
Cs· · ·O(3)$_c$	3.190 (6)	Cs· · ·O(2)	3.327 (6)

Mean Cs· · ·O 3.238

Angles, deg

Cs	O(2)$_g$	O(2)	O(3)$_f$	O(3)$_e$	O(3)$_d$	O(3)$_c$	O(1)$_b$
O(1)$_a$	52.5 (3)	129.3 (3)	112.6 (2)	67.9 (2)	57.3 (3)	119.9 (3)	177.9 (7)
O(3)$_c$	136.6 (1)	110.6 (1)	71.0 (1)	133.0 (2)	76.2 (2)		
O(3)$_e$	87.1 (2)	74.9 (2)	153.7 (2)				
O(2)	93.5 (2)						

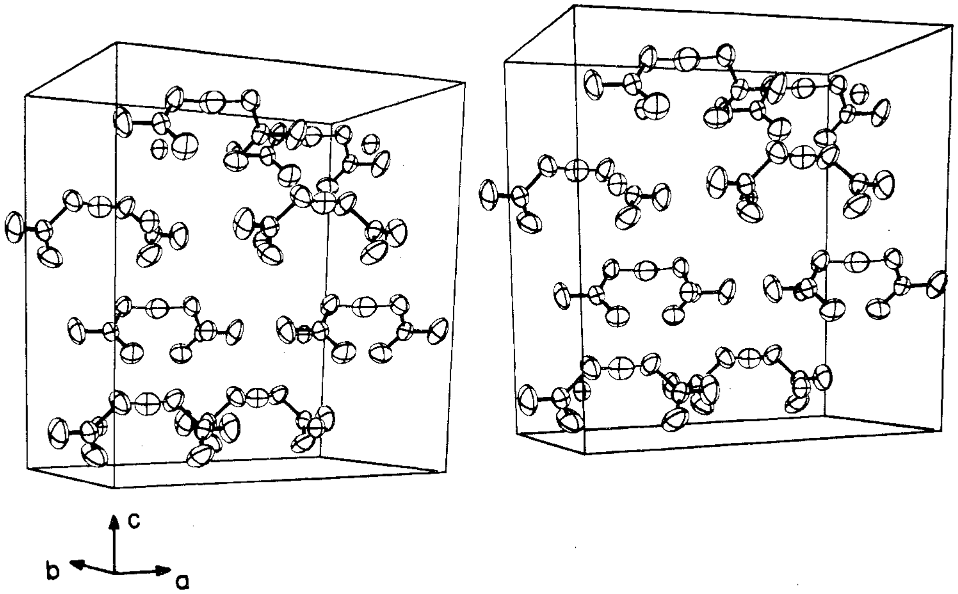

Figure 1. The stereoscopic drawing of the unit cell of $CsH(NO_3)_2$ which illustrates the packing of the Cs^+ and $H(NO_3)_2^-$ ions. For clarity, only half of the disordered sites of the hydrogen dinitrate ion are shown.

structure in which the O···O and O–H distances are in agreement with previous spectroscopic interpretations indicating the presence of a short and strong hydrogen bond. The hydrogen dinitrate ion is orientationally disordered into two positions and the heavy atom positions approximately superimpose on the Fourier maps to produce apparently ordered ones. The dihedral angle between the planes of two orientationally disordered nitrate groups is approximately 23° and the separations between the two atomic disordered sites (obtained from the least-squares refinement) are: N···N = 0.31 (2) Å, O(1)···0(1) = 0.44 (2) Å, and O(2)···O(3) = 0.55 (1) Å.

A stereoscopic view of the $CsH(NO_3)_2$ structure is given in Figure 1. The cesium ion has eight nearest neighbor oxygen atoms with distances less than 3.5 Å. The Cs^+···O distances and O···Cs^+···O angles are given in Table II. The cesium ion coordination polyhedron is presented in Figure 2. The geometry of an individual (ordered) hydrogen dinitrate ion is given in Figure 3 in addition to relevant distances and angles in Table III. The anion structure consists of two almost planar nitrate groups linked by a short hydrogen bond with O···O = 2.468 (8) Å. The hydrogen bond and nitrate group geometry in the cesium salt agrees fully with that found by Faithful and Wallwork[3] for one of the $H(NO_3)_2^-$ ions in the TPA salt in which a disorder also exists. However, differences occur in the respective orientations of the NO_3^- groups in the two salts. While the entire hydrogen dinitrate ion is planar in the TPA salt, the dihedral angle between nitrate group planes in the cesium salt is approximately 75°. A similar orientation (83°)

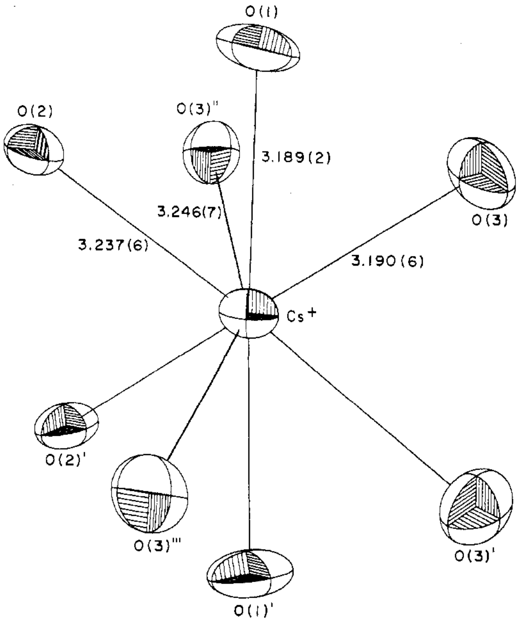

Figure 2. The coordination sphere around the cesium ion.

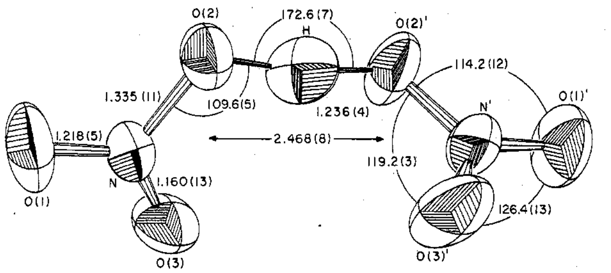

Figure 3. The geometry of an individual (ordered) hydrogen dinitrate ion in $CsH(NO_3)_2$. The $H(NO_3)_2^-$ ion has a nonplanar configuration where the two NO_3^- groups are related by symmetry through a twofold axis. The dihedral angle between the planes of two nitrate groups is 75.4 (4)°. The deviations of O(1), O(2), O(3), and N from a least-squares plane are 0.004 (7) Å, 0.003 (7) Å, 0.01 (2) Å, and −0.03 (2) Å, respectively.

Table III. Distances and Angles within the Hydrogen Dinitrate Ion

Bond Distances, Å[a]			
N–O(1)	1.218 (5)	O(2)–H	1.236 (4)
N–O(2)	1.335 (11)	O(2)· · ·O(2)$_h$[b]	2.468 (8)
N–O(3)	1.160 (13)		
Angles, deg			
O(1)–N–O(2)	114.2 (12)	N–O(2)–H	109.6 (5)
O(1)–N–O(3)	126.4 (13)	O(2)–H–O(2)$_h$[b]	172.6 (7)
O(2)–N–O(3)	119.2 (3)		
Short Nonbonded O· · ·O Distances, Å			
O(1)· · ·O(2)	2.144 (8)	O(2)· · ·O(3)$_h$[b]	3.119 (3)
O(1)· · ·O(3)	2.123 (12)	O(3)· · ·O(3)$_h$[b]	3.323 (10)
O(2)· · ·O(3)	2.152 (3)		

[a] The distances are uncorrected for thermal motion. [b] See symmetry operations given in Table II.

of the nearest neighbor NO_3^- groups occurs in the structure of the related compound $NH_4NO_3(HNO_3)_2$.[18] This may suggest that the particular $H(NO_3)_2^-$ configuration in the cesium salt is not only a result of the crystal packing effects but that both planar and nearly orthogonal NO_3^- group configurations correspond to energetically favorable configurations of the isolated ion. The covalent bond distances and angles within the NO_3^- group are in good agreement with those obtained for $TPAH(NO_3)_2$ and $NH_4NO_3(HNO_3)_2$. The N–O bond lengths for instance may be compared with those derived for the same ion in a *different* configuration in the TPA salt viz. 1.21 and 1.16 Å for the nonhydrogen bonded N–O bonds and 1.30 Å for that which is hydrogen bonded. The corresponding distances in the cesium salt are 1.218 (5), 1.160 (13), and 1.335 (11) Å. The thermal ellipsoids of the oxygen atoms are prolate in all cases and the direction of their largest principal axis is roughly perpendicular to the N–O bond which may indicate large amplitude motion of the NO_3^- groups.

Data for the hydrogen bond geometry are given in Table III and illustrated in Figure 3. The O···O distance is very short [2.468 (8) Å], being much shorter than the other nonbonded O···O distances between two nitrate groups and considerably less than we previously reported in the x-ray study[2] in which it was assumed that the $H(NO_3)_2^-$ groups were completely ordered. The hydrogen bond appears to be symmetric and slightly bent, the O–H–O angle being 172.6 (8) Å. The bridging hydrogen atom appears to be located on the twofold axis but of course our results do not preclude a disordered arrangement with two closely spaced sites. There is, however, no indication of this as would be indicated by elongation along the bond of the thermal ellipsoid of the bridging hydrogen atom. Indeed the thermal ellipsoid is not far from spherical in shape, but of course it does not correspond to the true motion of the hydrogen in the field of two static oxygen atoms. This relative motion is perhaps best described by the "riding model".[19] The rms displacement $[R^2(H)_{\|} - R^2(O)_{\|}]^{1/2}$, where $R_{\|}$ is the rms component of the total displacement of the bridging hydrogen atom along the O–H bond, is 0.196 Å. However, the perpendicular rms displacements $[R^2(H)_{\perp} - R^2(O)_{\perp}]^{1/2}$ are not real numbers. This does not necessarily imply that the temperature factors are inaccurate but rather suggests that the riding model is not the most appropriate one to describe the "perpendicular motion" at least in the present case where the NO_3^- groups seem to be undergoing large amplitude librational motion. The validity of the riding model (as far as the parallel motion is considered), as well the assumption of a single minimum hydrogen bond, can be checked by comparison with the spectroscopic data available in the literature. The value 0.196 Å for the rms displacement of the hydrogen atom along the O–H bond leads to a derived frequency of 440 cm^{-1} for ν_{OH}, using the relationship[20] $\nu = 16.8/m\langle r^2 \rangle$ where ν is in reciprocal centimeters, m in atomic mass units, and $\langle r^2 \rangle$ in ångströms squared, which is in good agreement with the values obtained from infrared spectroscopy (~500 cm^{-1})[2] and inelastic neutron scattering[9] (450 ± 20 cm^{-1}). Finally, it should be pointed out that the geometry found for the hydrogen dinitrate ion in $[Rh(py)_4Cl_2]H(NO_3)_2$ and $[(CH_3)_4N]H(NO_3)_2$ is remarkably similar to that reported in the x-ray study of $CsH(NO_3)_2$. In the light of the present results, it is quite likely that in those two salts the hydrogen dinitrate ion has the same configuration and is disordered in much the same way as in the cesium salt. Therefore it is possible that in all the cases, as was first deduced by Gillard and Ugo[7] from the ir spectra, the hydrogen dinitrate ion should be described as two nitrate groups linked by a very short and possibly symmetric hydrogen bond.

Acknowledgment. One of us (J.R.) has been supported by a program exchange grant from the National Science Foundation and the Centre National de La Recherche Scientifique which are gratefully acknowledged. J.R. is also grateful to the Argonne National Laboratory Chemistry Division for partial support.

Registry No. $CsH(NO_3)_2$, 35280-89-8; $H(NO_3)_2^-$, 12273-37-9.

Supplementary Material Available: A listing of structure factors for $CsH(NO_3)_2$ (2 pages). Ordering information is given on any current masthead page.

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The Molecular Structure and Absolute Configuration of *cis*-Dichloro[(*S*)-methyl *p*-tolyl sulfoxide][(*R*)-styrene]platinum(II)

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Received April 9, 1976 AIC60273C

The molecular structure and absolute configuration of the predominant diastereomer of *cis*-dichloro[(*S*)-methyl *p*-tolyl sulfoxide][styrene]platinum(II), $Cl_2[CH_3(O)S(C_6H_4CH_3)]Pt(C_6H_5HC{=}CH_2)$, have been determined from three-dimensional x-ray data collected by counter methods. The molecule crystallizes in the orthorhombic space group $P2_12_12_1$ with $Z = 4$. The unit cell dimensions are $a = 11.633$ (4), $b = 15.055$ (5), and $c = 9.776$ (3) Å. The structure has been refined by full-matrix least-squares techniques on F, using 2414 unique reflections for which $F^2 > 0$, to a final agreement factor of 0.0292. The complex adopts a square-planar coordination geometry, with the double bond of the coordinated olefin at an angle of 77.6 (6)° to the coordination plane. The absolute configuration at the asymmetric carbon atom which is formed upon olefin coordination has been determined by the Bijvoet method to be *R*.

Introduction

The absolute configurations of asymmetric metal–olefin complexes have been studied as a means of elucidating the causes of stereospecific nucleophilic additions to coordinated olefinic ligands.[1,2] Thus, a substituted olefin $H_2C{=}CHR$ such as styrene, $R = C_6H_5$, can coordinate to a metal atom through either of two enantiotopic faces. Upon coordination the substituted carbon atom of the olefin becomes a site of asymmetry. If the metal complex contains an additional enantiomeric ligand, two diastereomers may be formed, not necessarily in equal amounts. If the formation of one diastereomer is favored to a significant extent, then the asymmetric environment so provided may cause nucleophilic attack upon the olefin to be stereospecific, thus resulting in an asymmetric synthesis. In order that an understanding of the reaction mechanism may be reached, a knowledge of the absolute configuration of the coordinated olefin is essential.[2,3] Some progress in this field has recently been realized.[4]

Another area of interest in this laboratory is the study of the effects of the spatial distributions of asymmetric sites on the circular dichroism spectra of transition metal complexes.[5,6] The title complex has two asymmetric sites, the chiral sulfur atom of *S* absolute configuration and the asymmetric carbon atom of the olefin ligand. We report here the crystal structure and absolute configuration of the predominant diastereomer of the complex *cis*-dichloro[(*S*)-methyl *p*-tolyl sulfoxide][styrene]platinum(II).

Experimental Section

Pale yellow crystals of *cis*-dichloro[(*S*)-methyl *p*-tolyl sulfoxide][styrene]platinum(II), $Cl_2[CH_3(O)S(C_6H_4CH_3)]Pt(C_6H_5HC{=}CH_2)$, were kindly supplied by B. Bosnich and H. Boucher. Preliminary Weissenberg and precession photography showed the crystals to be orthorhombic with Laue symmetry *mmm*. The systematic absences observed, $h00$ for h odd, $0k0$ for k odd, and $00l$ for l odd, unambiguously determine the space group to be $P2_12_12_1$, D_2^4, No. 19.[7]

The crystal chosen for data collection was of approximate dimensions 0.04 × 0.06 × 0.19 mm. It was carefully measured on a microscope fitted with a filar eyepiece to facilitate an absorption correction. Ten faces of the forms {010}, {110}, and {011} were identified by optical goniometry. The crystal was mounted on a Picker FACS-1 computer-controlled diffractometer with [001] offset approximately 10° from coincidence with the spindle axis. Cell constants and an orientation matrix were obtained from a least-squares refinement of 26 intense, carefully centered reflections with $15 < 2\theta < 48°$. Prefiltered Cu radiation was used, λ 1.540 56 Å, at 20°C. Crystal data are given in Table I.

Table I. Crystal Data

$C_{16}H_{18}Cl_2OPtS$
$a = 11.633$ (4) A
$b = 15.055$ (5) A
$c = 9.776$ (3) A
$d(\text{obsd})^a = 2.04$ (1) g cm^{-3}
$\mu = 189.9$ cm^{-1} for Cu Kα_1

Fw 524.38
Space group $P2_12_12_1$
$Z = 4$
Cell vol = 1712.1 A^3
$d(\text{calcd}) = 2.03$ g cm^{-3}

[a] By neutral buoyancy in aqueous ZnI_2.

Table II. Experimental Conditions for Data Collection

Radiation: Cu Kα, Ni foil prefilter, 0.018 mm
Takeoff angle: 1.8° (gives 90% of maximum Bragg intensity)
Aperture: 4 mm × 4 mm, 32 cm from crystal
Data collected: *hkl* and $hk\bar{l}$, for $0 < 2\theta < 110°$
Scan: θ–2θ, range 1.2° corrected for dispersion, at 1° min^{-1}
Background: 20 s stationary crystal, stationary counter at limits of scan
Standards: six recorded every 200 observations; 002, $00\bar{2}$, 040, $0\bar{4}0$, 200, $\bar{2}00$

The conditions used for data collection are summarized in Table II. Several ω scans of intense, low-angle reflections were recorded as a check on crystal mosaicity. The average width at half-height was 0.08°. Measurement of standard reflections over the course of data collection and an examination of crystal mosaicity at the end showed no significant decomposition had occurred.

The intensity data were processed as previously described.[6] The value for p, the "ignorance" factor, was determined to be 0.01 from a statistical examination of the variations in the standard reflections.[8,9] An absorption correction was applied to all the data.[10] The maximum and minimum transmission coefficients were 0.424 and 0.232, respectively. As a check on the quality of the data, the 2539 reflections collected included 75 pairs of reflections for the equivalent forms *hk*0