charge distribution are listed in Table III for both molecules.

Discussion

It is interesting to compare several of the parameters in Table III with the similar and recently available results for other molecules. Several results of interest are listed in Table IV. The experimentally determined anisotropies of the magnetic susceptibility in the molecular plane ($\chi_{zz} - \chi_{yy}$) show that the sulfur-containing rings present less asymmetry in their response to the magnetic field than the corresponding oxygen-containing rings. It is also evident that the values of $\chi_{zz} - {}^1/_2(\chi_{xx} + \chi_{yy})$ decrease in magnitude progressively from benzene to ethylene oxide in Table IV. The values of $\chi_{zz} - {}^1/_2(\chi_{xx} + \chi_{yy})$ may be considered a criterion for aromaticity or delocalization of the perpendicular π electrons in these ring systems. In other words,[8] ring currents can be excited by magnetic fields perpendicular to the ring with increasing ease in the series from ethylene oxide to benzene in Table IV.

It is also interesting to compare the molecular quadrupole moments in these ring compounds. The out-of-plane (x) molecular quadrupole moments are negative in all molecules in Table IV except ethylene oxide. This indicates the presence of the electrons above and below the molecular plane. Furthermore, the in-plane moments perpendicular to the symmetry axes (y) are all positive. These positive moments are due to the partially shielded protons in the outer regions of the molecule along the y axes. These simple correlations may be of help in estimating molecular quadrupole moments along the principal inertial axes in other molecules.

The out-of-plane values for the second moment of the charge distribution in these molecules are also listed in Table IV. These values seem to correlate as expected with the number and type of heavy atoms in the plane and the number of out-of-plane protons.

Acknowledgment. The support of the National Science Foundation is gratefully acknowledged.

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The Reduction of Benzene by Hydrated Electrons in γ-Ray Irradiated Alkaline Solutions[1]

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Contribution from the Chemistry Division, Argonne National Laboratory,
Argonne, Illinois 60439. *Received December 30, 1968*

Abstract: Hydrated electrons convert benzene into 1,4-cyclohexadiene (1,4-C_6H_8) which is further reduced to cyclohexene (C_6H_{10}) and cyclohexane (C_6H_{12}). In addition dimeric products progress from $C_{12}H_{14}$ to $C_{12}H_{22}$ as reduction proceeds. The hydrated electrons were produced by the γ-ray irradiation of pH 13 solutions saturated with H_2 at 1500 psi pressure.

Since the hydrated electron (e_{aq}^-) was identified in 1962 its rate of reaction with some 400 organic compounds has been measured. However, only in comparatively few cases were the products identified. The hydrated electrons, H atoms, and OH radicals formed by ionizing radiations in aqueous solutions react with benzene to form complex combinations of partially reduced and oxidized compounds including biphenyl and phenol.[2–6] In a recent pulse radiolysis study of aqueous benzene at pH 3 with methanol as the OH radical scavenger, the cyclohexadienyl radical, resulting from H-atom addition, was identified. This radical disappears in a second-order diffusion-controlled reaction.[7] Product analysis of the aqueous solutions was not attempted. In the radiolytic reduction of gaseous benzene in hydrogen and argon mixtures the H atom produces the cyclohexadienyl radical intermediate and 1,4-C_6H_8 and 1,3-C_6H_8 as the final products. When the hydroxyl radical reacts with aqueous C_6H_6 it too adds to the ring and forms the hydroxycyclohexadienyl radical, C_6H_6OH.[8] In the present work, complications introduced by the H atoms and OH radicals were avoided by irradiating C_6H_6 solutions at 100 atm of H_2 pressure at pH 13. Under these conditions H and OH change into e_{aq}^- by the reactions

$$OH + H_2 = H_2O + H$$

$$H + OH^- = e_{aq}^- + H_2O$$

At pH 13 the equilibrium ratio $[(e_{aq}^-)/(H)]_{equil} = 2300$ [calculated from $k(e_{aq}^- + H_2O) = 16\ M^{-1}\ sec^{-1}$ and $k(H + OH) = 2 \times 10^7\ M^{-1}\ sec^{-1}$]. Because the rate constant ratio $k(e_{aq}^- + C_6H_6)/k(H + C_6H_6) = 0.01$, the e_{aq}^- reaction dominates H-atom addition to benzene by a factor of 23.[9] Consequently with less than 40

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(1) Based on work performed under the auspices of the U. S. Atomic Energy Commission.

(2) M. Daniels, G. Scholes, and J. Weiss, *J. Chem. Soc.*, 832 (1956).

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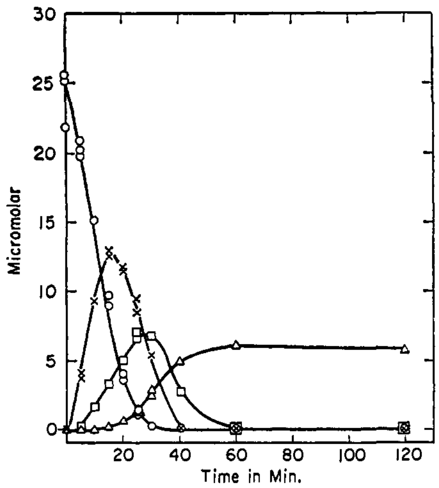

Figure 1. Effect of time of irradiation on volatile product formation in 25 μM C_6H_6, 0.13 M H_2 in 0.1 N NaOH: ○, C_6H_6; ×, C_6H_8; □, C_6H_{10}; △, C_6H_{12}. Dose rate = 7.7 × 10^{19} eV/l. min.

μM C_6H_6 and 0.08 M H_2 at pH 13, the amount of reaction of benzene with OH is less than 2% of that with e_{aq}^-. Our objective was to determine the usefulness of e_{aq}^- in studying organic reduction mechanisms under these conditions.

Results

Measurements by a combination gas chromatograph–mass spectrometer[10] showed that aqueous benzene solutions are stable in 0.08 M H_2 at pH 13 for periods up to 68 hr at room temperature. On irradiation with Co^{60} γ rays the benzene was reduced after a brief but variable induction period caused by trace impurities. The reduction proceeded to 1,4-cyclohexadiene, cyclohexene, and finally cyclohexane. After short irradiations only benzene and cyclohexadiene were detected.

The effect of dose on the loss of C_6H_6 and the formation of 1,4-C_6H_8, C_6H_{10}, and C_6H_{12} are given in Figure 1. In this 25 μM C_6H_6 run we show the expected early appearance of 1,4-C_6H_8 and its conversion into C_6H_{10}, and finally after 60 min irradiation, C_6H_{12} is the only volatile product left. We assume that 75% of the C_6H_6 forms dimers and other high molecular weight products.

When the irradiations were done in heavy water, extensive deuteration of benzene and its reduction products resulted. A 40 μM solution of benzene in D_2O at pD 13.4 and 0.08 M H_2 was given a radiation dose of 23.7 × 10^{20} eV/l. The chromatogram of the gas is shown in Figure 2. Fractionation of each species according to degree of deuteration was shown by a series of fast-scan mass spectra across each peak. The resulting data are summarized in Table I.

Less volatile products were also investigated by the combination gas chromatograph–mass spectrometer system. Ten milliliters of solution 30 μM in benzene, 0.08 M in H_2 at pH 13, was irradiated with a dose of 3.7 × 10^{21} eV/l. and extracted with 0.2 ml of benzene. After evaporation of benzene the residue was distilled onto the open tube column and fractionated at 130–140°. Figure 3 represents the chromatogram obtained. Twenty components were identified by the combination system. These represent dimeric species configurations and degrees of hydrogenation. The data are summarized in Table II. The fully saturated $C_{12}H_{22}$ at mass 166 was not observed in this experiment, but it appeared in samples given a high radiation dose.

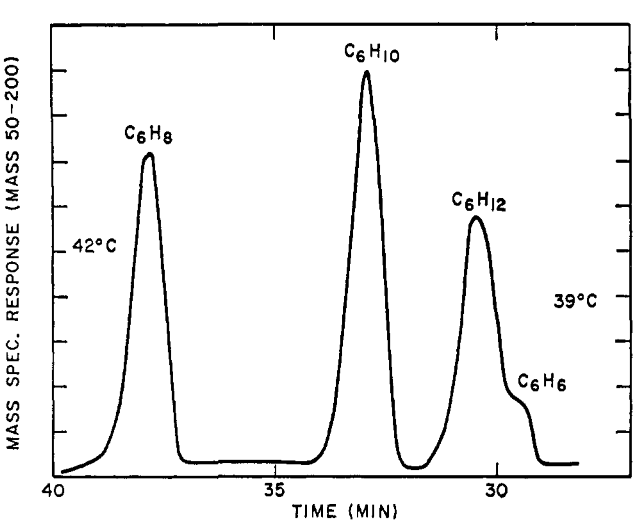

Figure 2. Separation of volatile fractions by a capillary column and identification by mass spectrometry: 40 μM C_6H_6, 0.08 M H_2 in 0.1 N NaOD irradiated 10 min at a dose rate of 2.37 × 10^{20} eV/l. min; open tube column 91.5 m long and 0.25 mm i.d. programmed over the range 39–42°.

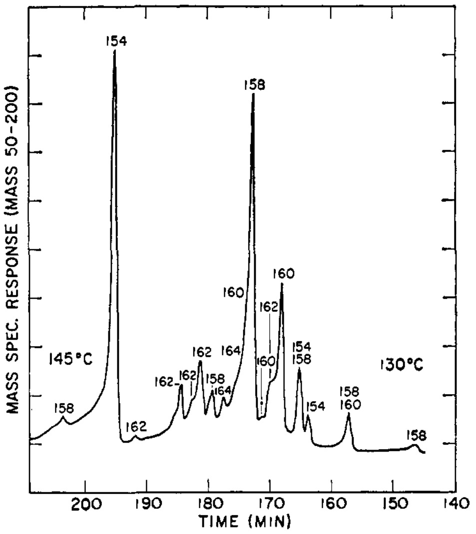

Figure 3. Fractionation of dimers by a capillary column and identification by mass spectrometry: 30 μM C_6H_6, 0.08 M H_2 in 0.1 N NaOH; dose = 3.7 × 10^{21} eV/l; programmed temperature range = 130–140°.

Table I. Deuteration of Benzene and Its Reduction Products[a]

Compound	Most Abundant	Most D
Benzene	C_6H_6	$C_6H_2D_4$
Cyclohexadiene	$C_6H_6D_2$ + $C_6H_5D_3$	$C_6H_3D_5$
Cyclohexene	$C_6H_7D_3$ + $C_6H_6D_4$	$C_6H_5D_5$
Cyclohexane	$C_6H_8D_4$ + $C_6H_7D_5$	$C_3H_6D_6$

[a] 40 μM C_6H_6; pD 13.4; 0.08 M H_2; γ-ray dose of 23.7 × 10^{20} eV/l.

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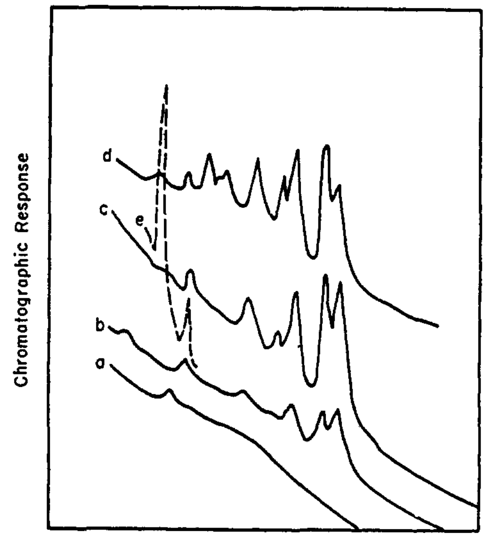


Figure 4. Chromatographic separation of dimers at 110°. Effect of time of irradiation on dimer formation and disappearance in irradiated 40 μM C_6H_6, 0.08 M H_2, in 0.1 N NaOH. Dose rate = 2.37×10^{20} eV/l. min: (a) 0 and 4 min, (b) 6 min, (c) 10 min, (d) 20 min, and (e) 40 min. $C_{12}H_{22}$ elutes first; the highly unsaturated ones last ($C_{12}H_{10}$ elutes well beyond the last bands shown).

Dimers also form at early stages of the irradiation. Figure 4 shows that after 20 min of irradiation all the dimers from $C_{12}H_{14}$ to $C_{12}H_{22}$ are present. Many are already present after 6 min. Except for $C_{12}H_{22}$ these products have not been positively identified, but from the mass spectral analysis discussed above, we know that they fall into this range. Furthermore, we established that biphenyl, $C_{12}H_{10}$, elutes last at the far right beyond the double peak. After 40 min the bands on the right disappear leaving but one major band, that of dicyclohexyl, on the extreme left.

Table II. Dimers Formed in Benzene Solutions at pH 13[a]

Mass no.	Dimer formula	No. of isomers
154	$C_{12}H_{10}$	3
156	$C_{12}H_{12}$	0
158	$C_{12}H_{14}$	6
160	$C_{12}H_{16}$	4
162	$C_{12}H_{18}$	5
164	$C_{12}H_{20}$	2
166	$C_{12}H_{22}$	0

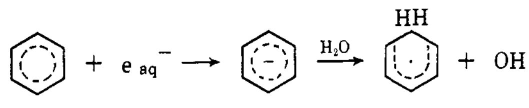
[a] 0.08 M H_2; γ-ray dose of 3.70×10^{21} eV/l.

Discussion

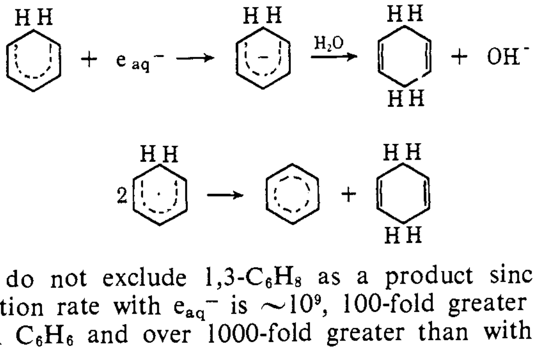
Benzene is reduced by solvated electrons in NH_3[11,12] and amines[13,14] as well as electrochemically.[15,16] The principal primary product is 1,4-C_6H_8 which forms to the exclusion of its isomer, 1,3-C_6H_8. According to current theory the e_{sol}^- adds to benzene, forming a negative ion which has been identified by ultraviolet and esr spectroscopy.[12,17] This negative ion then abstracts a proton from NH_3 or more readily from proton donors such as alcohols added to NH_3.[18] The resulting radical then adds an e_{sol}^- and the protonation is repeated forming C_6H_8. In recent pulse radiolysis studies Sauer and Ward[7] report a transient cyclohexadienyl radical, C_6H_7, in aqueous solutions. Its strong ultraviolet absorption band centered at 311 nm has a molar extinction coefficient of 5.4×10^3 M^{-1} cm^{-1}. This radical decays by a second-order process in aqueous solutions with a rate constant of 1.8×10^9 M^{-1} sec^{-1}.

In our work with low C_6H_6 concentrations, 0.08 M H_2 at pH 13, e_{aq}^- reactions are dominant in the primary and in some secondary reactions. We pointed out above that under our conditions the $(e_{aq}^-)/(H)$ ratio equals 2300, and even though the relative rate constant ratio, $k(e_{aq}^- + C_6H_6)/k(H + C_6H_6) = 0.01$, the e_{aq}^- reaction dominates H addition by a factor of 23. But these are only for the initial conditions, and as radicals and new products appear their relative reactivities too must be considered. With our conditions favoring e_{aq}^- reactions in mind, and accepting the facts regarding negative ion and $C_6H_7\cdot$ formation, we outline a mechanism that accounts for the main features of our results.

Monomer Products. 1,4-C_6H_8, C_6H_{10}, and C_6H_{12} successively form as the radiolysis proceeds, and in D_2O, C_6H_6 becomes deuterated (Figure 2 and Table I). After electron attachment we postulate protonation as follows.



Subsequent reactions of the $C_6H_7\cdot$ radical may be with e_{aq}^- or by disproportionation but not by reaction with C_6H_6.[7,19] And while the e_{aq}^- rate constant is not known we believe $k(e_{aq}^- + C_6H_7\cdot) \sim 10^{10}$ M^{-1} sec^{-1}, possibly a factor of 10 higher than the rate constant for bimolecular disappearance. Thus we have



We do not exclude 1,3-C_6H_8 as a product since its reaction rate with e_{aq}^- is $\sim 10^9$, 100-fold greater than with C_6H_6 and over 1000-fold greater than with 1,4-C_6H_8 or C_6H_{10}.[19] Consequently 1,3-C_6H_8 once formed would be rapidly transformed into C_6H_{10} or into a dimer. Because of the low reactivity of e_{aq}^- toward 1,4-C_6H_8 and C_6H_{10} relative to H atoms, their reduction to C_6H_{12}, the final product, is first by H-atom addition.

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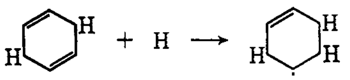
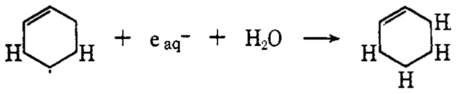
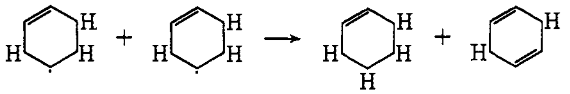


But because the ratio $(e_{aq}^-)/(H) = 2300$, the e_{aq}^- reaction with this $C_6H_9\cdot$ radical dominates the H reaction



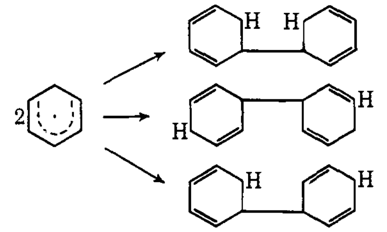
However, disproportionation forming cyclohexene and C_6H_8 also occurs.



Through similar processes the inert C_6H_{12} finally forms.

In D_2O extensive deuteration takes place. The reformation of C_6H_6 in the disproportionation reaction will, of course, produce deuterated benzene.

Dimerization of the radicals $C_6H_7\cdot$, $C_6H_9\cdot$, and $C_6H_{11}\cdot$ and their possible combinations account for the products $C_{12}H_{14}\cdots C_{12}H_{22}$, but Figure 4 shows that the unsaturated dimers gradually hydrogenate forming $C_{12}H_{22}$ as the final product (see discussion of Figure 4 under Results). Reduction of the dimeric compounds will be by H atoms instead of by e_{aq}^- unless conjugated double bonds are present.[19] The biphenyl (mass 154) shown in Figure 4 probably originates by dehydrogenation of $C_{12}H_{14}$ during analysis since it is not found in the lower temperature (110°) chromatographic separation of Figure 4. These dimeric reactions are



Since only three dimers of the C_6H_7–C_6H_7 type are possible, we believe that of the six $C_{12}H_{14}$'s reported in Table II, three of the type C_6H_5–C_6H_9 form by dehydrogenation of C_6H_7–C_6H_9 during fractionation at 130–140°. Reduction of the two conjugated double bond products would be rapid and dominated by e_{aq}^-.

Because of its highly selective reducing properties, e_{aq}^- is active only in the stages involving C_6H_6 and products such as 1,3-C_6H_8 and the conjugated dimers. We believe that e_{aq}^- has considerable potential as an interesting new reducing agent especially in alkaline solutions.

Experimental Section

Aqueous solutions pressurized to 1500 or to 2400 psi of H_2 were irradiated at Co^{60} γ-ray dose rates of 1.71×10^{20} eV/l. min, corresponding to 17.6 μM e_{aq}^-/min unless otherwise specified. Dose rates were determined by the Fricke dosimeter using $G(Fe^{3+}) = 15.6$. After irradiation benzene and the volatile products were extracted from the solution with air or hydrogen or by fractional distillation in a vacuum system; the nonvolatile products were extracted with zone-refined benzene and the extracts analyzed by gas chromatography, by time-of-flight mass spectrometry,[10] or by a combination of the two.

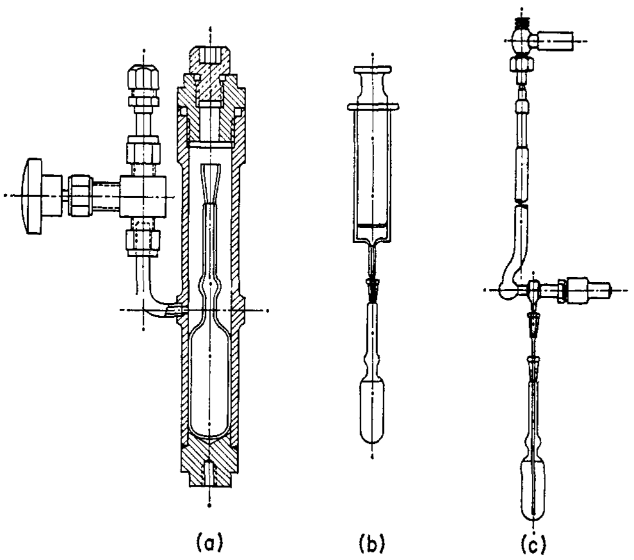


Figure 5. Pressure cell illustrating technique of extracting volatile products: (a) assembled cell, (b) cell-syringe assembly used in extracting volatile products for gas chromatography, (c) cell-sample storage assembly used for time-of-flight mass spectrometer analysis.

Materials and Solutions. The materials used were as follows: triply distilled D_2O (99.7% D) and H_2O saturated with hydrogen and containing less than 10^{-7} M O_2; C_6H_6, Litton, zone-refined 99.999% pure; H_2, Matheson, 3500 psi prepurified 99.95% passed through a palladium catalyst (Engelhard deoxo purifier, Model D10-2500); NaOH, 20 M stock solution prepared from Baker reagent grade NaOH; NaOD, 5 M stock solution prepared by dissolving 99.999% pure metallic sodium in H_2-saturated D_2O; 1,3-cyclohexadiene, Aldrich Chemical Co.; 1,4-cyclohexadiene, Farchan Research Laboratory.

Saturated aqueous benzene solutions of about 20 mM were obtained by adding 0.2 ml of C_6H_6 to 50 ml of H_2-saturated triply distilled water in a 100-ml syringe. The solution was saturated with C_6H_6 by shaking with 20 cc of H_2 for a few minutes. The excess benzene and H_2 were then discharged. Our benzene concentrations are based on a molar extinction coefficient of 185 M^{-1} cm^{-1} at the main 240-nm absorption band. This extinction coefficient was obtained for a standard solution made by dissolving C_6H_6 in ethanol in syringes and then diluting with water under conditions in which there was no gas phase.

High-Pressure Cell. Our pressure cell assembly is shown in Figure 5. The outer jacket consists of a stainless-steel cylinder 6 in. in height and 1 in. in diameter. Its side arm has a valve which is connected to a high-vacuum line, vented with an external bubble tube to indicate flow rate. The Pyrex irradiation cell shown in Figure 5b is inserted through the $^3/_4$-in. hole in the top. The top consists of two parts, one having a $^3/_4$-in. entry, the other a $^3/_8$-in. one. Their caps are sealed with O rings. Before filling the irradiation cell with solution, we pump it down to about 1 mm pressure, pressurize it to 500 psi H_2, and then vent the gas to atmospheric pressure, letting a slow steady stream of hydrogen flow through the bubble tube to indicate a positive H_2 pressure inside the system. When the small $^3/_8$-in. cap is taken off the pressure cell, the hydrogen flows through it, thereby preventing entry of air. The cell is then filled with the test solution. Next the vessel is capped and pressurized to 1500 psi, then shaken for 10 min. The irradiation cell itself consists of a 12-ml Pyrex body with a stem with an enlarged section to keep the solution from bubbling out when the pressure is released. Its top also has a 5/20 female joint.

Product Analysis. The volatile hydrocarbons consist of C_6H_6, 1,4-C_6H_8, C_6H_{10}, and C_6H_{12}. There was no evidence of any other low molecular weight hydrocarbons. We separated these compounds on an 8-ft column at room temperature,[7] and also on a 300-ft, 0.01-in. capillary column.[10] Each product including the 1,3-C_6H_8 and 1,4-C_6H_8 could be satisfactorily separated. From calibration curves run for each product on solutions of known concentration under conditions identical with those used for the irradiation, we attempted a quantitative analysis. We found that the peak height *vs.* concentration was linear for all the compounds. They were also identified by mass spectrometry.

For mass spectrometric measurements the samples were introduced into the evacuated tube, shown in Figure 5c, by dipping the capillary tip into the sample and opening the Teflon stopcock. Aliquots of the gas phase above the aqueous solution were taken through the metal valve at the top. After condensation in a liquid nitrogen trap, the H_2 was pumped away and the sample was warmed and examined with the mass spectrometer. For detailed analyses, samples were distilled into an open tube column (91.5 m long and 0.25 mm i.d., coated with Apiezon L). The column was developed in a stream of He and detected and analyzed by the time-of-flight mass spectrometer.[10]

The nonvolatile but benzene-extractable dimers consisting of the group of compounds $C_{12}H_{14}\cdots C_{12}H_{22}$ were separated on a Perkin-Elmer 900 gas chromatograph programmed 10 min at 80°, then 2°/min to 110° on a 1.5% OV-17 on Chromosorb W column. They were also separated at 130–140° on a stainless steel open tube column 91.5 m long and 0.25 mm i.d., coated with Apiezon L. The dimers were then detected and identified by mass spectrometry.[10]

Acknowledgments. We are grateful to Drs. Weldon Brown and Myran Sauer for their help in the separation and identification of products and to Miss P. Walsh and Messrs. Robert M. Clarke and Leon P. Moore for technical assistance. We also thank Mr. Edmund E. Klocek for the design of the high-pressure cells.

The Molecular and Crystal Structure of (±)-*trans*-1,2-Cyclohexanedicarboxylic Acid

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Received January 22, 1969

Abstract: The crystal structure of (±)-*trans*-1,2-cyclohexanedicarboxylic acid has been determined. The unit cell is monoclinic with $a = 5.65$, $b = 13.34$, $c = 11.04$ Å; $\beta = 113°\ 16'$. The space group is C2/c with four molecules per unit cell. The crystal structure was determined by the application of close-packing criteria in the hypothesis that the molecules are assembled in hydrogen-bonded rows and that the C_2 symmetry of the molecule is retained as a crystallographic symmetry element. In the crystalline state the preferred conformation of carboxyl groups is diequatorial. The mode of packing of rows is discussed.

Each molecule of the (±)-*trans*-1,2-cyclohexanedicarboxylic acid possesses two asymmetric centers having the same chirality. As far as the conformation is concerned, two conformers are possible; *i.e.*, either both carboxyls are in axial positions, or both are equatorial. Studies of rate of acid-catalyzed esterification led Smith and Byrne[1] to describe that the diaxial form would predominate in the *trans* acid since the carboxyl groups are more widely separated. Other authors[2,3] confirmed that the *trans* acid should exist as diaxial from considerations of the dissociation constants as compared with those of the *cis* isomer and from the fact that the intermolecular polymeric anhydride tends to be formed from the *trans* acid. On the other hand, it has been suggested[4] that the *trans* acid exists as diequatorial on the basis of acidity constants of a series of analogous alicyclic compounds. In order to establish the conformation in the solid state for the *trans*-1,2-cyclohexanedicarboxylic acid, we have examined single crystals of this compound by means of X-ray diffraction.

Experimental Section

Crystals suitable for X-ray analysis were obtained by slow evaporation of aqueous solution (mp 232–233°). On the basis of Weissenberg's photograph taken with Cu Kα radiation, the crystals were found to belong to the monoclinic system. The extinctions of hkl reflections for $h + k = 2n + 1$ and $h0l$ for $h = 2n + 1$, and $l = 2n + 1$ indicate two possible space groups: the centrosymmetric C2/c, or the acentric Cc. A small crystal was carefully centered on a Picker automatical four-circle diffractometer equipped with a PDP-8 digital computer. The determination of the lattice constants was carried out by a least-squares refinement of the setting angles of 12 reflections using Cu Kα radiation (λ 1.5418 Å). The parameters obtained are reported in Table I. The density calculated, $d = 1.43$ g/cm^3, on the basis of the molecular weight (mol wt = 172.18) with assuming four molecules per unit cell agreed reasonably with the experimental value of $d = 1.41$ g/cm^3 obtained by flotation method. The intensity data were collected using the 2θ scan mode of the diffractometer with Ni-filtered Cu Kα radiation. The take-off angle of the tube was 3.0° and a counter aperture, 4.0 × 4.0 mm, was placed 30 cm from the crystal. A scan angle of 1.50° was found to be sufficient for all reflections over the range (0–130°) of 2θ examined. The scan speed was 1°/min. Two stationary-crystal–stationary-counter background counts of 10 sec were taken at each end of each scan.

Table I. Unit Cell Dimensions

(±)-*trans*-1,2-Cyclohexanedicarboxylic acid: $C_8O_4H_{12}$	
Mol wt = 172.18	F(000) = 368
Monoclinic, space group C2/c	
$a = 5.65 \pm 0.01$ Å	
$b = 13.34 \pm 0.03$ Å	$\beta = 113°\ 16' \pm 10'$
$c = 11.04 \pm 0.03$ Å	
$D_{X-R} = 1.43$ g/cm^3	$Z = 4$

Structure Determination and Refinement

The structure of the (±)-*trans*-1,2-cyclohexanedicarboxylic acid was determined by a relatively straightforward application of close-packing criteria.[5] At the

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