should be noted that the gas-phase reaction coordinate in Figure 6 is a typical reaction coordinate, i.e., gas-phase ion–molecule reaction kinetics studies show that very many reactions of the type $A^- + B \rightarrow C^- + D$ proceed via a transition state whose energy lies *below* that of the reactants. This is a consequence of the attractive interactions between the ion $A^-$ and the molecule B which most often are large enough to compensate the energy increase attendant the formation of the transition state. This is the case for all reported reactions which proceed in the gas phase at collision rates.[4,5,26]

The cause for faster rate in the aprotic solvent was deduced above, it is the lesser sensitivity of the aprotic solvent to the increased size of the transition-state ion, i.e., solvation exothermicity decreases with an increase of ion size in both protic and aprotic solvents but less so for the aprotic solvent.

It is often stated[2,3,25] that the (presumed) stronger interaction of the aprotic solvents with the larger negative ion is due to the large polarizability of the ion and the solvent molecule and the effect of "mutual polarization". The data in Tables I and IV clearly show that the interaction of the larger ion with the aprotic solvent is not stronger but weaker than that with the smaller ion. The analysis above also showed that the lesser sensitivity of the

aprotic solvent to an increase of negative ion radius is mostly due to the large size of the aprotic solvent molecule (which makes the inner-shell cluster large). Thus the relationship with the large polarizability of the aprotic molecule is only accidental insofar that large size goes with large polarizability.[27] Finally, the theoretical calculations of the bonding of $Cl^-$ to $Me_2SO$ in Table III clearly show that neither the polarizability nor the dispersion terms are of decisive importance to the bonding of the negative ion with the dipolar aprotic solvent.[27,28]

The very strong interaction of the positive ion ($M^+$) with aprotic solvents (Tables I, III, and IV) also has synthetic utility in negative ion reactions like eq 1 since it promotes the solubility of the salt MX in the solvent ($X^- = Cl^-$ in eq 1) and by complexing with the ion $M^+$ it prevents the formation of $M^+X^-$ ion pairs. The formation of an ion pair represents a stabilization of the ion $X^-$ and thus acts to reduce its reactivity.

**Registry No.** $Me_2SO$, 67-68-5; $CH_3Br$, 74-83-9.

(26) Nibering, N. N. M. In "Kinetics of Ion Molecule Reactions"; Ausloos, P., Ed.; Plenum Press: New York, 1979.

(27) The notion that the polarizability is a decisive term in the interaction of large ions with large ligands is a basic part of the Hard and Soft Lewis Acid and Base Theory (HSAB) (Pearson[28]), which is presumed to apply to 1:1 complexes. However, the experimental data on which the theory is based involve complexes in solution. Proper consideration of solvent effects significantly changes some of the rationalizations of HSAB.

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# Gas-Phase Structure of Dimethyl Peroxide

**Bernhard Haas and Heinz Oberhammer***

*Contribution from the Institut für Physikalische und Theoretische Chemie der Universität Tübingen, 7400 Tübingen, West Germany. Received January 23, 1984*

**Abstract:** The geometric structure of dimethyl peroxide, $CH_3O–OCH_3$, was studied by gas electron diffraction. The molecular intensities were analyzed by applying a large amplitude model with a double minimum potential for the O–O torsion. The following geometric parameters for the COOC skeleton were obtained: O–O = 1.457 (12) Å, O–C = 1.420 (7) Å, OOC = 105.2 (5)°, and dihedral angle $\theta$ = 119 (10)°. Distances and angles are $r_a$ values, and the dihedral angle corresponds to the minima of the potential function. Experimental uncertainties are based on $3\sigma$ values and include systematic errors due to the assumptions for vibrational amplitudes and the analytical expression for the potential function. A planar trans configuration of the COOC skeleton with large-amplitude torsional vibration must be rejected on the basis of the electron-diffraction data.

Molecular structures of peroxides have attracted much interest by experimentalists and theoreticians for the past decades. The structural feature of principal interest is the dihedral angle. For noncyclic peroxides dihedral angles from less than 90° (87.5° in $F_2O_2$[1]) to 180° have been determined. (In this connection only gas-phase structures are considered, since packing effects and intermolecular interactions may strongly affect this parameter.) This large range for the dihedral angle indicates that this parameter is a delicate balance between two opposing effects: (1) interaction between the oxygen lone pairs which favors a dihedral angle of about 90° (assuming sp hybridization for oxygen) and (2) repulsion between the substituents which tends to increase this angle.

For dimethyl peroxide experimental as well as theoretical studies produce rather controversial results for the dihedral angle. An early electron diffraction analysis by the visual method[2] reports an average value for the O–O and C–O bond lengths and the OOC angle, but no value for the dihedral angle. Two PES investigations interpret the splitting of the oxygen lone pair ionization potentials in terms of an exactly planar ($\theta = 180°$) or nearly planar ($\theta =$

170°) trans configuration. In the first study[3] the interpretation of the PES data is based on CNDO/2 calculations which predict a planar trans configuration, whereas in the latter study[4] the energy splitting of the lone pair orbitals is compared to those of other peroxides. Analysis of the IR and Raman spectra[5] and a normal coordinate analysis based on these data,[6] however, reject a planar trans ($D_{2h}$ symmetry) or cis ($C_{2v}$ symmetry) configuration and indicate $C_2$ symmetry. Microwave spectra for dimethyl peroxide have been recorded,[7,8] demonstrating a nonzero dipole moment, excluding a planar trans configuration. So far, these spectra have not been assigned. Semiempirical and ab initio molecular orbital calculations predict dihedral angles for dimethyl peroxide ranging from 96.5° (MINDO/2[9]) to 180° (CNDO/2[3] and ab initio[10]). The MINDO/3 method (110.7°[11]) and ab initio calculations with

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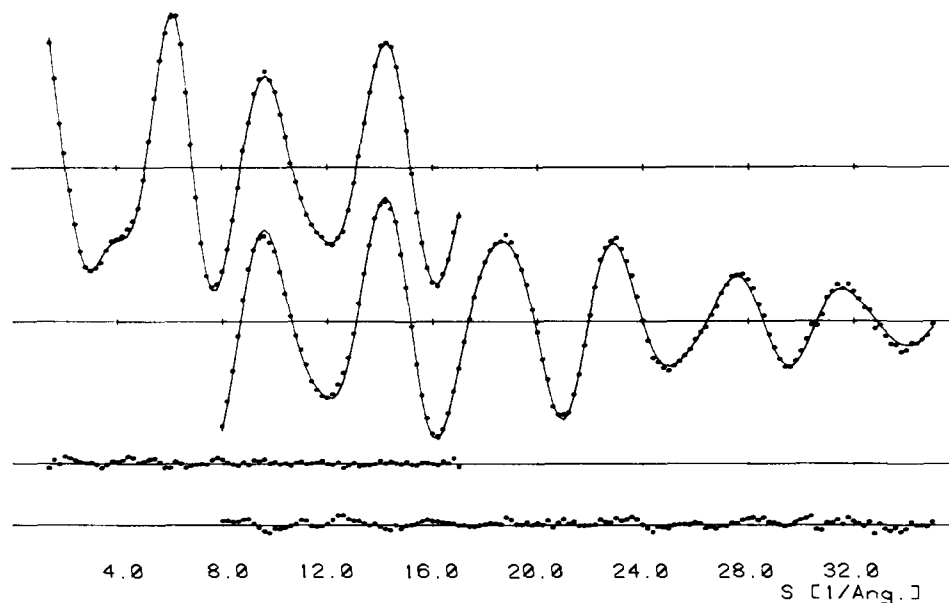(10) Bair, R. A.; Goddard, W. A., III *J. Am. Chem. Soc.* **1982**, *104*, 2719–2724.

**Figure 1.** Experimental (···) and calculated (—) molecular intensities and differences.

STO-2G (101.69° [12]) and 4-31G* (115.5° [13]) basis sets result in intermediate values. The strong dependence of this parameter on the semiempirical method or on the basis set used in the ab initio calculation is not surprising, considering that large changes of the dihedral angle result from small energy differences. According to the O–O torsional potential calculated in ref 10, the energy increases only by about 0.25 kcal mol$^{-1}$ between 180° and 120° dihedral angle (estimated from Figure 2 of ref 10). The aim of the present investigation is to study the structure of dimethyl peroxide by gas electron diffraction, and it is hoped that this method can unambiguously discriminate between a planar trans and a skewed configuration.

## Experimental Section

Dimethyl peroxide was synthesized according to literature methods.[14,15] The sample was purified by repeated trap-to-trap fractionation until no impurities could be detected in the gas IR spectrum which was recorded in a 10-cm cell at the vapor pressure corresponding to –30 °C (about 250 torr). The electron-diffraction intensities were recorded with the Balzers Gas Diffractograph KD-G2[16] at two camera distances (50 and 25 cm) and an accelerating voltage of about 60 kV. The sample was kept at –30 °C (same condition as used for the IR spectrum) and inlet system and nozzle were kept at room temperature. The gas flow was regulated with a needle valve. Exposure time was 6–10 s and 30–45 s for the long and short camera distances, respectively. The electron wavelength was determined from ZnO diffraction patterns. Two plates were selected for each camera distance and analyzed by the usual procedures.[17] Extraneous scattering, recorded without gas, was subtracted from the 25-cm data. The averaged molecular intensities are shown in Figure 1, and numerical values of the total scattering intensities in steps of $\Delta s = 0.2$ A$^{-1}$ are available as supplementary data.

## Structure Analysis

A rough estimate of the skeletal COOC parameters can be obtained from the radial distribution function (Figure 2): (O–O,C–O)$_{av}$ = 1.43 Å, C···O = 2.28 Å, C···C = 2.34 Å. From these

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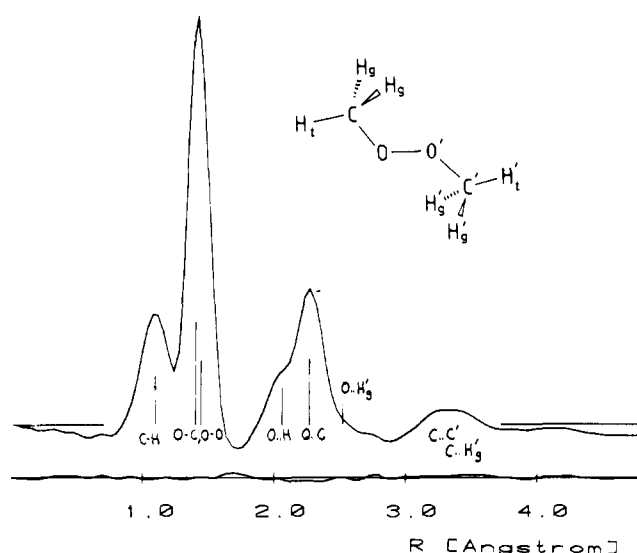
**Figure 2.** Experimental radial distribution function and difference curve.

distances we obtain OOC = 106° and dihedral angle COOC = 135°. Thus, the COOC skeleton is nonplanar, and the very broad peak corresponding to the C···C distance indicates a large-amplitude COOC torsional vibration. These starting parameters were refined in least-squares analyses by using two different molecular models. The first analysis was based on a rigid molecular model which implies the assumption of small-amplitude harmonic vibrations. A diagonal weight matrix was applied to the intensities[17] and scattering amplitudes and phases of Haase[18] were used. The methyl groups were assumed to possess $C_{3v}$ symmetry and to stagger the O–O bond. These assumptions are justified by the ab initio calculations of Gase and Boggs[13] which result in only slightly distorted methyl groups nearly staggering the O–O bond.

The angle distortion can be very well accounted for by a tilt angle of about 3° between the $C_3$ axis and the O–C bond direction. The electron-diffraction intensities, however, are not sensitive toward such a tilt angle, and assuming either 0° or 3° results in the same skeletal parameters. Since the O–O and C–O bond lengths are very similar, high correlations do not allow a simultaneous refinement of these bond lengths and their amplitudes. Therefore, these amplitudes were constrained to the spectroscopic values which were calculated from the force field of ref

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6148 *J. Am. Chem. Soc., Vol. 106, No. 21, 1984*

*Haas and Oberhammer*

**Table I.** Results of Least-Squares Analysis[c]

(a) Geometric Parameters and Trans Barrier (kcal mol⁻¹)

| | | | |
|---|---|---|---|
| O–O | 1.457 (12) | HCH | 110.1 (7)° |
| O–C | 1.420 (7) | $\theta$ | 119 (4)° |
| C–H | 1.099 (4) | $V_t$ | 0.25 (+0.25, –0.10) |
| OOC | 105.2 (5) | | |

(b) Vibrational Amplitudes (without H···H amplitudes)

| | e.d. | spectr. | | e.d. |
|---|---|---|---|---|
| O–O | 0.050 (4)[a] | 0.050 | O'···H$_t$ | 0.100[b] |
| C–O | 0.051 (4)[a] | 0.051 | O'···H$_g$ | 0.157 (21) |
| C–H | 0.083 (5) | 0.079 | C'···H$_t$ | 0.150[b] |
| O···C | 0.068 (4) | 0.071 | C'···H$_g$ | 0.250[b] |
| C···C | 0.065 (10)[a] | 0.065 | | |
| O···H | 0.098 (8) | 0.104 | | |

(c) Agreement Factors

$$R_{50} = 3.4\% \qquad R_{25} = 6.4\%$$

[a] Not refined. The estimated uncertainty is included in the error analysis. The C···C amplitude does not include COOC torsion. [b] Not refined. [c] Large-amplitude model with potential function given by eq 1. Distances and angles are $r_a$ values, $\theta$ corresponds to the minimum of the potential function. For error limits see text; atom numbering is indicated in Figure 2.

6. This force field does not include force constants for the methyl torsions or COOC torsion, and vibrational amplitudes for torsion-independent distances only are meaningful. Further assumptions concerning vibrational amplitudes are evident from Table I. With these constraints six geometric parameters (O–O, C–O, C–H, OOC, HCH, COOC) and four vibrational amplitudes for a rigid molecular model were refined. The dihedral angle derived in this analysis (135 (5)°) is an effective value due to the large-amplitude COOC torsion. For the C···C vibrational amplitude a value of 0.256 (62) Å is derived. All other parameters agree within their standard deviations with the values in Table I which were derived by using the second model. In this case the parameters of a large-amplitude model, with the COOC torsion separated from the other molecular vibrations,[19] were refined. The methyl torsions, although large-amplitude vibrations as well, have only a small effect on the molecular intensities. For the COOC torsion a double minimum potential of the general shape

$$V(\phi) = V_t\{(\phi/\phi_e)^2 - 1\}^2 \qquad (1)$$

was assumed with $\phi = 180 - \theta$; $V_t$ the torsional barrier in the trans conformation and $\phi_e$ corresponds to the minimum of the potential energy. This expression cannot describe the actual potential in the vicinity of the cis barrier ($\phi = 180°$), which is estimated to be higher than 10 kcal mol⁻¹, but it is very similar to a fourier expansion for $0 < \phi < 90°$. Since electron-diffraction data are not sensitive to states above 1 kcal mol⁻¹, eq 1 is felt adequate for this analysis. Although this expression is by no means unique, it has the advantage of containing the relevant parameters $V_t$ and $\phi_e$ explicitly. The molecular intensities are calculated by using[19]

$$M(s) = \int M(s,\phi)P(\phi)\,d\phi$$

with $P(\phi) = N \exp(-V(\phi)/RT)$

In this analysis the C···C amplitude was constrained to the rigid frame value derived from the spectroscopic data. In a series of refinements with different but fixed values of $V_t$, six geometric parameters and three vibrational amplitudes (see Table I) were refined. Only two correlation coefficients had values larger than 0.5: CO/OO = –0.77 and OOC/HCH = 0.52. The best fit of the molecular intensities was obtained for $V_t$ = 0.25 kcal mol⁻¹, assuming that gas and nozzle temperature were equal. The optimum value for $V_t$ depends on the assumption for the C···C amplitude. Estimating a systematic error of 0.01 Å for the

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**Table II.** Geometric Parameters of Dimethyl Peroxide from Experimental and Theoretical Methods

| | O–O | C–O | OOC | $\theta$ |
|---|---|---|---|---|
| e.d., this study | 1.457 (12) | 1.420 (7) | 105.2 (5) | 119 (10) |
| e.d.[a] | 1.44 (2) | 1.44 (2) | 105 (3) | |
| ab initio, GVB + CI[b] | 1.450 | 1.444 | 104.1 | 180 |
| ab initio, 4-21 G*[c] | 1.411 | 1.422 | 105.4 | 115.5 |
| ab initio, STO 2G[d] | 1.3822 | 1.4704 | 101.69 | 100.65 |
| MINDO/3[e] | 1.391 | 1.360 | 113.1 | 110.7 |
| W.-H.[f] | 1.490 | 1.428 | 104.0 | 118 |

[a] Electron diffraction study of ref 2. [b] Reference 10. [c] Reference 13. [d] Reference 12. [e] Reference 11. [f] Westheimer–Hendrickson calculations, ref 20.

spectroscopic value of this amplitude, an upper limit of 0.50 and a lower limit of 0.15 kcal mol⁻¹ is obtained. The results of the least-squares analysis using a large amplitude model with the potential function of eq 1 are summarized in Table I. The experimental uncertainties are $3\sigma$ values and include systematic errors of 0.004 Å for the C–O and O–O vibrational amplitudes. Additional least-squares refinements with a double-minimum potential given by

$$V(\phi) = V_t\{(\phi/\phi_e)^3 - 1\}^2 \qquad (2)$$

resulted in a dihedral angle of 112 (5)°, whereas all other parameters remained practically unchanged. Equation 2 differs from eq 1 mainly in the steepness toward smaller dihedral angles (see Figure 3).

These least-squares refinements using various models demonstrate that the value derived for the dihedral angle strongly depends on the molecular model used in the analysis: 135 (5)° for a rigid model and 119 (4)° or 112 (5)° for a large-amplitude model with use of potential functions of eq 1 or eq 2. The sum of the errors squared in the least-squares analysis is smallest for potential function 1, and it increases by 2% for eq 2 and by 6% for the rigid model. As pointed out above, high quality ab initio calculations including electron correlation[10] result in a flat single minimum potential (see curve c in Figure 3) which can be approximated by $V = a\phi^4$ ($a = 2 \times 10^{-8}$, $\phi$ in degrees). Such a potential function, however, is in very poor agreement with the experimental molecular intensities. The sum of the errors squared increases by 24%, which is a strong increase, considering that only distances $r > 2.5$ Å are affected by the potential function. Thus, our electron-diffraction data are not compatible with a planar skeleton performing a large-amplitude torsional vibration.

## Discussion

The present electron-diffraction study for dimethyl peroxide results in a nonplanar conformation of the COOC skeleton with a dihedral angle of 119 (4)° and a large-amplitude torsional vibration. Since the above experimental uncertainty does not include systematic errors due to the choice of the potential function, a more realistic value for this uncertainty is likely to be 10°. The present result is in agreement with the IR and Raman spectra and with the microwave experiment, but it disagrees with the interpretation of the PES spectra which yielded a planar or nearly planar skeleton.[3,4] This apparent discrepancy can be explained in the following manner: at room temperature the majority of the molecules are in vibrational states above the trans barrier (0.25 kcal mol⁻¹) and thus, for these molecules, the initial state for the ionization process corresponds to a pseudoplanar configuration. The splitting of the lone pair energy levels for the vibrationally excited states (1.90 eV) should be close to the value expected for a planar configuration. As pointed out by Rademacher,[20] both ionization bands have shoulders with an energy difference of about 1.1 eV, corresponding to a dihedral angle of approximately 125°. These shoulders would then correspond to ionization from vi-

(20) Rademacher, P., private communication.

**Table III.** Comparison of Geometric Parameters and Trans Barriers (kcal mol$^{-1}$) of Some Peroxides

| | O–O | O–C/O–Si | OOC/OOSi | $\theta$ | $V_t$ |
|---|---|---|---|---|---|
| HO–OH[a] | 1.464 | | | 121.6 | 1.1[b] |
| CH$_3$O–OH[c] | 1.443 | 1.437 | 105.7 | 114 | 0.25 |
| CH$_3$O–OCH$_3$[d] | 1.457 (12) | 1.420 (7) | 105.2 (5) | 119 (10) | 0.25 (+0.25, –0.10) |
| CF$_3$O–OCF$_3$[e] | 1.419 (20) | 1.399 (9) | 107.2 (12) | 123 (4) | |
| *t*-BuO–O-*t*-Bu[f] | 1.480[g] | 1.460 (9) | 103.9 (12) | 166 (3) | |
| Me$_3$SiO–OSiMe$_3$[f] | 1.481 (8) | 1.681 (3) | 106.6 (14) | 144 (6) | |

[a] Reference 21. [b] Reference 22. [c] Reference 23. [d] This study. [e] Reference 24. [f] Reference 25. [g] This parameter was not refined in the analysis.
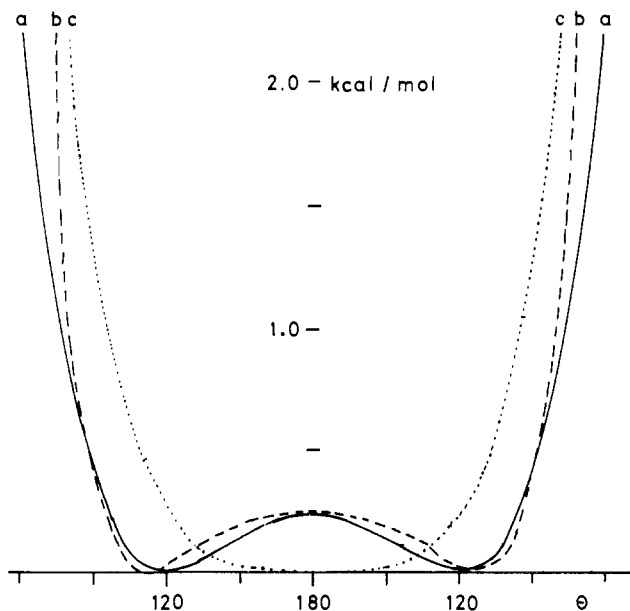


**Figure 3.** Potential functions for COOC torsion: (a) eq 1 with $V_t = 0.25$ kcal/mol and $\phi_e = 61°$ ($\theta_e = 119°$); (b) eq 2 with $V_t = 0.25$ kcal/mol and $\phi_e = 68°$ ($\theta_e = 112°$); (c) $V(\phi) = a\phi^4$ with $a = 2 \times 10^{-8}$ and $\phi$ in degrees.

brational states below the barrier.

Theoretical calculations at different levels (force field, semi-empirical, and ab initio) predict dihedral angles between 96.5° (MINDO/2[9]) and 180° (CNDO/2[3] and ab initio[10]). Investigations with optimization of all parameters of the COOC skeleton are summarized in Table II. The ab initio calculation of Gase and Boggs[13] (115.5°), MINDO/3[11] (110.7°), and Westheimer–Hendrickson calculations[20] (118°) predict dihedral angles in agreement with our experiment (119 (10)°). The reason for the failure of the ab initio calculations of Bair and Goddard[10] to predict the correct configuration of dimethyl peroxide is not at all obvious. These calculations use a larger basis set than Gase and Boggs, including polarization functions for oxygen and configuration interaction. While Gase and Boggs, however, optimize all geometric parameters, Bair and Goddard optimize only the COOC skeletal parameters, constraining the geometric parameters for the CH$_3$ groups. The full structure optimization results in some distortion of the methyl groups and, expecting that these distortions depend on the dihedral angle, i.e., increase with decreasing dihedral angle, they can possibly affect the general shape of the potential. Only small energy differences (<1 kcal mol$^{-1}$) are required to change the single minimum potential in Figure 3 into a double minimum potential close to the experimental result.

A comparison of the bond lengths predicted by the various theoretical methods shows that both high quality ab initio cal-

culations[10,13] are in good agreement with the experiment. The O–O bond length derived with the 4-21G* basis set is shorter by 0.04 Å than the electron-diffraction value. The predictions of the STO-2G calculations are rather poor giving also the wrong sequence of the distances (O–O < C–O instead of O–O > C–O). All theoretical calculations, except MINDO/3 and STO-2G, give an excellent prediction for the OOC bond angle.

Table III compares geometric parameters of some peroxides. The O–O bond length clearly correlates with the electronegativity of the substituent. The longest bond is observed in Me$_3$SiO–OSiMe$_3$ (1.481 (8) Å[21]) and the shortest bond in CF$_3$O–OCF$_3$ (1.419 (20) Å[22]). The peroxide bond lengths in hydrogen peroxide and dimethyl peroxide are equal within their error limits. The O–C and O–Si bonds in the peroxides are longer than the bonds in the analogous ethers (O–C = 1.410 (3) Å in (CH$_3$)$_2$O[26] or 1.369 (4) Å in (CF$_3$)$_2$O,[27] O–Si = 1.634 (2) Å in (SiH$_3$)$_2$O[28]). The OOC and OOSi angles for all peroxides in Table III are very similar. The most interesting structural parameter for these peroxides, the dihedral angle, is around 120° for substituents such as H, CH$_3$, and CF$_3$. From this we conclude that steric effects are not important in determining the structures of these compounds. There is, however, a substituent effect on the trans barrier which is smaller in CH$_3$OOCH$_3$ and CH$_3$OOH (0.25 kcal mol$^{-1}$) than in H$_2$O$_2$ (1.1 kcal mol$^{-1}$). The increase in the dihedral angles in *t*-BuO–O-*t*-Bu and Me$_3$SiO–OSiMe$_3$ can be attributed to steric repulsions between the bulky substituents.

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