dimethylgold bromide by reaction with hydrogen chloride in the presence of excess lithium bromide.[2] $(CH_3)_2AuBr$ shows characteristic infrared absorptions at 3000, 2955, 2908, 1405, 1229, 1198, 810, 722, and 530 cm^{-1} (Hostaflon, Nujol), and a proton magnetic resonance signal at -82.3 cps relative to TMS as an internal standard in CCl_4 solution (Varian A 60, 60 Mc, 35°).

Equimolar amounts of dimethylgold bromide and sodium trimethylsiloxide were found to react in diethyl ether solution at room temperature with formation of a sodium bromide precipitate. After filtration

$$(CH_3)_2AuBr + NaOSi(CH_3)_3 \longrightarrow NaBr + \underset{\text{I}}{(CH_3)_2AuOSi(CH_3)_3}$$

and evaporation of the solvent under vacuum a colorless crystalline material was obtained which could be sublimed *in vacuo* at 40° under 0.001 mm pressure. The sublimate, mp 39–40°, is soluble in aprotic organic solvents and stable to dry air, but rather sensitive to moisture. Combustion analysis confirmed the composition $(CH_3)_5AuOSi$, but cryoscopic molecular weight determinations in benzene as a solvent proved the compound to exist as a *dimer*.

Anal. Calcd: C, 18.97; H, 4.74; mol wt, 632. Found: C, 18.65; H, 4.50; mol wt, 603.

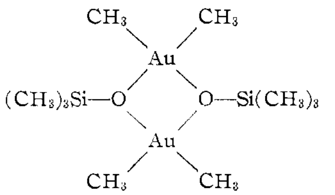
The colorless melt of I is decomposed at 135° with formation of a bright gold mirror at the walls of the tube. The compound gives rise to two singlet signals in its ^1H nmr spectrum, the areas of which were found to be in the relative ratio 3:2: δCH_3Au -57.7 cps, δCH_3Si -8.5 cps (CCl_4 solution). The $(CH_3)_3Si$ signal is accompanied by two small satellite signals due to $^1H-^{29}Si$ coupling with $J(^1H-C-^{29}Si) = 6.5$ cps. In the infrared spectral region 1350–400 cm^{-1} only a small number of bands were detected, and their assignments are given in Table I. These spectra along with the observed diamagnetism of the compound (nmr) are consistent with an inorganic skeleton containing the gold atoms in a square-planar dsp^2 hybridization state. For I therefore a bridged four-membered ring structure is suggested

TABLE I

INFRARED SPECTRUM OF TRIMETHYLSILOXYDIMETHYLGOLD[a]

Cm^{-1}	Assignment	Cm^{-1}	Assignment
1300 w	$\delta_{as}CH_3(Si)$	805 sh	$\rho CH_3(Au)$
1258 s		720 sh	
1245 s		680 m	$\nu_{as}SiC_3$
1210 s	$\delta_{as}CH_3(Au)$	620 w	ν_sSiC_3
910 vs	$\nu Si-O$	588 m	$\nu_{as}AuC_2$
882 vs		577 m	
835 vs	$\rho CH_3(Si)$	478 vs	$\nu Au-O$
748 s		440 sh	

[a] Perkin-Elmer 337, KBr windows, Nujol solution.



As has recently been shown, oxygen-bridged structures are not uncommon in heterosiloxane chemistry,[1] but I is the first example with the heteroatom in a nontetrahedral state of bonding.[3]

Attempts to prepare tris(trimethylsiloxy)gold from $AuBr_3$ and 3 moles of $NaOSi(CH_3)_3$ were successful only at temperatures below $-30°$. A yellow crystalline compound was obtained on recrystallization from diethyl ether, which slowly decomposed above $-20°$. The initial product showed the following infrared absorptions: 1260, 1245, 875 (νSiO), 840, 752, 681, 638, 502 (νAuO), 470 (νAuO). In the ^1H nmr spectrum two signals appeared at δCH_3Si -12.8 and -20.2 cps (ratio 1:2). However, the presence of impurities could not entirely be excluded, as suggested from small additional signals. Again a structure similar to that of I with bridging and terminal siloxy groups is proposed for this compound.[3,4]

[3] H. Schmidbaur, *Chem. Ber.*, **96**, 2692 (1963); **97**, 255, 836, 952 (1964).

[4] H. Schmidbaur and F. Schindler, *Angew. Chem.*, **77**, 865 (1965); H. Schmidbaur, *ibid.*, **77**, 169 (1965); H. Schmidbaur and B. Armer, *ibid.*, **78**, 305 (1966).

CONTRIBUTION FROM THE DEPARTMENT OF CHEMISTRY,
MICHAEL FARADAY LABORATORIES, NORTHERN ILLINOIS
UNIVERSITY, DEKALB, ILLINOIS 60115

A General Formulation for the Evaluation of Group Overlap Intergals

BY WALTER A. YERANOS

Received June 15, 1966

During the past few years the semiempirical method of Wolfsberg and Helmholz[1] has gained tremendous momentum in the explanation of the magnetic and optical properties of the coordination compounds of the transition metal ions.[2]

As is well known, one of the computational problems encountered in this method is the evaluation of the overlap integrals between the symmetry-adapted functions of the central ion and the ligands, as well as between the ligands themselves. Fortunately, when the ligands lie on the cartesian coordinate system which defines the molecular symmetry, extremely simple techniques[3,4] can be devised for the reduction of the group overlap integrals to *scaled* diatomic overlaps. The latter can then be evaluated either from published tables[5] or by the use of digital computers.[6] Unfortu-

[1] M. Wolfsberg and L. Helmholz, *J. Chem. Phys.*, **20**, 837 (1952).

[2] C. J. Ballhausen, "An Introduction to Ligand Field Theory," McGraw-Hill Book Co. Inc., New York, N. Y., 1962.

[3] H. D. Bedon, S. M. Horner, and S. Y. Tyree, *Inorg. Chem.*, **3**, 647 (1964).

[4] C. J. Ballhausen and H. B. Gray, "Molecular Orbital Theory," W. A. Benjamin, Inc., New York, N. Y., 1965.

[5] (a) R. S. Mulliken, C. A. Rieke, D. Orloff, and H. Orloff, *J. Chem. Phys.*, **17**, 1248 (1949); (b) H. H. Jaffé and G. O. Doak, *ibid.*, **21**, 196 (1953); (c) H. H. Jaffé, *ibid.*, **21**, 258 (1953); (d) J. L. Roberts and H. H. Jaffé, *ibid.*, **27**, 883 (1957); (e) L. Leifer, F. A. Cotton, and J. R. Leto, *ibid.*, **28**, 364, 1253 (1958); (f) D. P. Craig, A. Maccoll, R. S. Nyholm, L. E. Orgel, and L. E. Sutton, *J. Chem. Soc.*, 354 (1954).

[6] W. A. Yeranos, Moment Integral Calculations, Q.C.P.E. Program 82, Quantum Chemistry Program Exchange, Indiana University, Bloomington, Ind. 47401.

nately, however, when the molecule under investigation has a complex geometry, matters are not as simple.

The aim of the present note is to present a general formulation by which *any* metal–ligand or ligand–ligand overlap of the transition metal complexes can be easily evaluated. The formulation takes into consideration the *arbitrary orientation* of the coordinate systems of both centers and in this respect goes beyond the recent formulation discussed by Kettle.[7] Its use necessitates the knowledge of the three Eulerian angles needed to bring each center into the usual "alignment" required for standard diatomic integral calculations.[5,6] Figure 1 depicts the successive rotations needed to bring about such an "alignment." It should be stressed that on *both* coordinate systems the sequence of rotations is the same. First, there is a rotation through an angle γ about the original z axis, Z. This is then followed by a rotation through an angle β about the new y axis, Y', and finally the system is rotated through an angle α about the z axis resulting from the previous transformation, Z''. The final position of the redescribed coordinate system is given by X_0, Y_0, and Z_0. It is *important*, however, to note that in right-handed coordinate systems clockwise rotations give positive angles (Figure 1a), while in left-handed coordinate systems anticlockwise rotations give positive angles (Figure 1b).

The basic mathematical development of the following relationships has been discussed elsewhere[8,9] and consequently shall not be repeated here. Suffice it to say, however, that we have considered only the ns, np, and nd orbitals. Furthermore, since the ns orbitals are invariant under any rotational transformation, we shall only give the redescriptions of the np and nd orbitals. Thus if we define

$$\xi \equiv \cos(\beta/2)$$

and

$$\eta \equiv -\sin(\beta/2)$$

then for the p orbitals, we have

$$\mathrm{p}_x = \{\xi^2 \cos(\alpha+\gamma) - \eta^2 \cos(\alpha-\gamma)\}\mathrm{p}_{x_0} + \{2\xi\eta\cos\gamma\}\mathrm{p}_{z_0} + \{\xi^2 \sin(\alpha+\gamma) - \eta^2 \sin(\alpha-\gamma)\}\mathrm{p}_{y_0}$$

$$\mathrm{p}_z = \{-2\xi\eta\cos\alpha\}\mathrm{p}_{x_0} + \{\xi^2 - \eta^2\}\mathrm{p}_{z_0} - \{2\xi\eta\sin\alpha\}\mathrm{p}_{y_0}$$

$$\mathrm{p}_y = \{-\xi^2 \sin(\alpha+\gamma) - \eta^2 \sin(\alpha-\gamma)\}\mathrm{p}_{x_0} - \{2\xi\eta\sin\gamma\}\mathrm{p}_{z_0} + \{\xi^2\cos(\alpha+\gamma) + \eta^2\cos(\alpha-\gamma)\}\mathrm{p}_{y_0}$$

and for the d orbitals

$$\mathrm{d}_{x^2-y^2} = \{\xi^4\cos(2\alpha+2\gamma) + \eta^4\cos(2\alpha-2\gamma)\}\mathrm{d}_{x_0^2-y_0^2} + \{2\xi^3\eta\cos(\alpha+2\gamma) - 2\xi\eta^3 \times \cos(\alpha-2\gamma)\}\mathrm{d}_{x_0z_0} + \{2\sqrt{3}\xi^2\eta^2\cos 2\gamma\}\mathrm{d}_{z_0^2} + \{2\xi^3\eta\sin(\alpha+2\gamma) - 2\xi\eta^3\sin(\alpha-2\gamma)\}\mathrm{d}_{y_0z_0} + \{\xi^4\sin(2\alpha+2\gamma) + \eta^4\sin(2\alpha-2\gamma)\}\mathrm{d}_{x_0y_0}$$

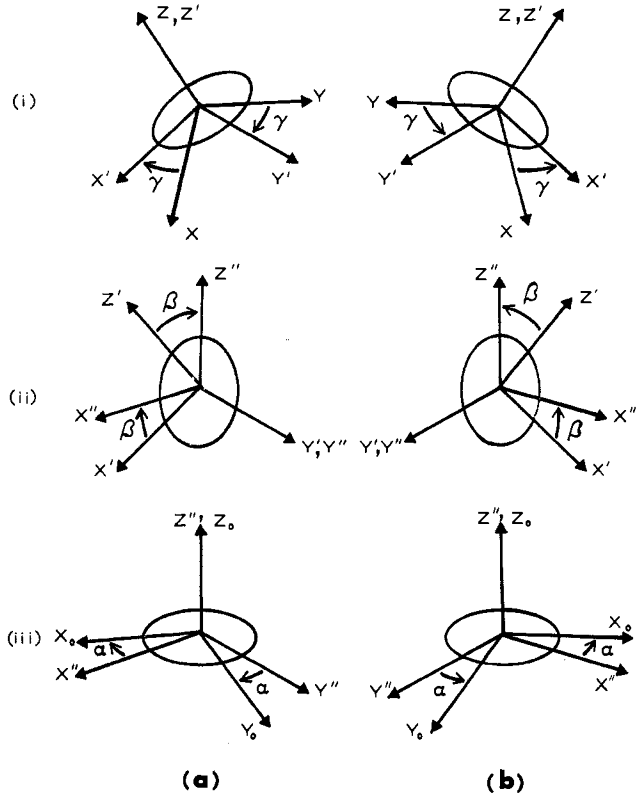


Figure 1.—Eulerian angle rotations for "proper" alignment: (a) for right-handed coordinate systems; (b) for left-handed coordinate systems.

$$\mathrm{d}_{xz} = \{2\xi\eta^3\cos(2\alpha-\gamma) - 2\xi^3\eta\cos(2\alpha+\gamma)\}\mathrm{d}_{x_0^2-y_0^2} + \{[\xi^4 - 3\xi^2\eta^2]\cos(\alpha+\gamma) - [3\xi^2\eta^2 - \eta^4]\cos(\alpha-\gamma)\}\mathrm{d}_{x_0z_0} + \{2\sqrt{3}[\xi^3\eta - \xi\eta^3] \times \cos\gamma\}\mathrm{d}_{z_0^2} + \{[\xi^4 - 3\xi^2\eta^2]\sin(\alpha+\gamma) - [3\xi^2\eta^2 - \eta^4]\sin(\alpha-\gamma)\}\mathrm{d}_{y_0z_0} - \{2\xi^3\eta\sin(2\alpha+\gamma) - 2\xi\eta^3\sin(2\alpha-\gamma)\}\mathrm{d}_{x_0y_0}$$

$$\mathrm{d}_{z^2} = \{\sqrt{12}\xi^2\eta^2\cos 2\alpha\}\mathrm{d}_{x_0^2-y_0^2} - \{\sqrt{12}[\xi^3\eta - \xi\eta^3]\cos\alpha\}\mathrm{d}_{x_0z_0} + \{\xi^4 - 4\xi^2\eta^2 + \eta^4\}\mathrm{d}_{z_0^2} - \{\sqrt{12}[\xi^3\eta - \xi\eta^3]\sin\alpha\}\mathrm{d}_{y_0z_0} + \{\sqrt{12}\xi^2\eta^2\sin 2\alpha\}\mathrm{d}_{x_0y_0}$$

$$\mathrm{d}_{yz} = \{2\xi\eta^3\sin(2\alpha-\gamma) + 2\xi^3\eta\sin(2\alpha+\gamma)\}\mathrm{d}_{x_0^2-y_0^2} - \{[\xi^4 - 3\xi^2\eta^2]\sin(\alpha+\gamma) + [3\xi^2\eta^2 - \eta^4]\sin(\alpha-\gamma)\}\mathrm{d}_{x_0z_0} - \{2\sqrt{3}[\xi^3\eta - \xi\eta^3]\sin\gamma\}\mathrm{d}_{z_0^2} + \{[\xi^4 - 3\xi^2\eta^2] \times \cos(\alpha+\gamma) + [3\xi^2\eta^2 - \eta^4] \times \cos(\alpha-\gamma)\}\mathrm{d}_{y_0z_0} - \{2\xi^3\eta\cos(2\alpha+\gamma) + 2\xi\eta^3\cos(2\alpha-\gamma)\}\mathrm{d}_{x_0y_0}$$

---

(7) S. F. A. Kettle, *Inorg. Chem.*, **4**, 1821 (1965).

(8) J. L. Synge and B. A. Griffith, "Principles of Mechanics," McGraw-Hill Book Co., Inc., New York, N. Y., 1959.

(9) M. E. Rose, "Elementary Theory of Angular Momentum," John Wiley and Sons, Inc., London, 1957.

$$\mathrm{d}_{xy} = -\{\xi^4 \sin(2\alpha + 2\gamma) - \eta^4 \sin(2\alpha - 2\gamma)\}\mathrm{d}_{x_0{}^2-y_0{}^2} - \{2\xi^3\eta \times \sin(\alpha + 2\gamma) + 2\xi\eta^3 \sin(\alpha - 2\gamma)\}\mathrm{d}_{x_0z_0} - \{2\sqrt{3}\xi^2\eta^2 \sin 2\gamma\}\mathrm{d}_{z_0{}^2} + \{2\xi^3\eta \times \cos(\alpha + 2\gamma) + 2\xi\eta^3 \cos(\alpha - 2\gamma)\}\mathrm{d}_{y_0z_0} + \{\xi^4 \cos(2\alpha + 2\gamma) - \eta^4 \cos(2\alpha - 2\gamma)\}\mathrm{d}_{x_0y_0}$$

where the old functions described in the reoriented coordinate system are given in terms of the basis set p_{x_0}, p_{y_0}, etc. As an example, we take the familiar setup given by Figure 2, where

$$\xi \equiv \cos(3\pi/4) = -1/\sqrt{2}$$

and

$$\eta \equiv -\sin(3\pi/4) = -1/\sqrt{2}$$

An overlap of the following type

$$\langle \mathrm{d}_{x^2-y^2} | \sigma \rangle$$

is then found to be

$$\langle \mathrm{d}_{x^2-y^2} | \sigma \rangle \equiv -\frac{1}{2}\langle \mathrm{d}_{x_0{}^2-y_0{}^2} | \sigma \rangle + \frac{\sqrt{3}}{2}\langle \mathrm{d}_{z_0{}^2} | \sigma \rangle$$

Finally, there are two points which we wish to bring to the reader's attention.

The first deals with the proper definition of the "usual alignment" previously mentioned. When the two coordinate systems which describe the two-center integral belong to different handedness, the "usual alignment" culminates by the combination of coordinate systems given in Figure 3a, while if they belong to the same handedness it culminates by a similar combination as the one given in Figure 3b. It is instructive to note that if two orbitals $\psi(\mathrm{n'l'm'})$ and $\psi(\mathrm{nlm})$ are defined on centers A and B, respectively, *any* two-center integral M evaluated in the combination given by Figure 3a is related to an equivalent value M′ obtained in the combination given by Figure 3b. That is

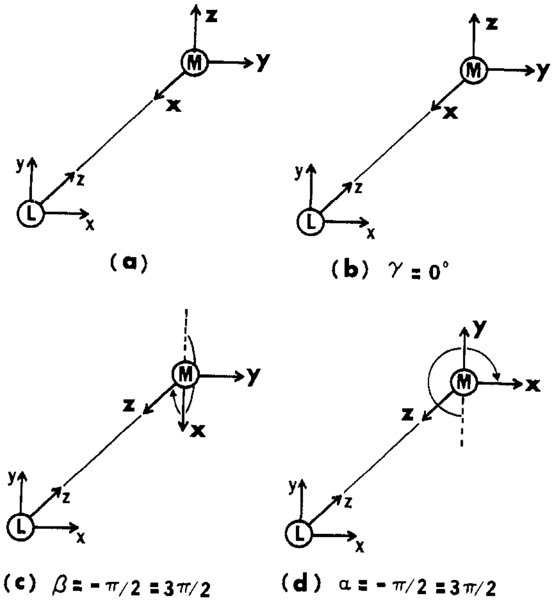
$$\mathrm{M'} = (-1)^{\mathrm{l+m}}\mathrm{M}$$

The last point should obviously be considered if ligand–ligand overlaps are to be evaluated from published tables.[6]

Another point which we wish to discuss is the fact that although the emphasis was put on overlap integral calculations, the formulas presented in this note can very well be used in *any* two-center calculations which involve coordinate reorientation. In particular, they can be used in moment[10] integral calculations involving integrals of the general type

$$\langle \psi(\mathrm{n'l'm'}) | X_i^{\alpha} Y_i^{\beta} Z_i^{\gamma} | \psi(\mathrm{nlm}) \rangle$$

where X_i, Y_i, and Z_i are the coordinates which describe the position of the ith electron. The origin of this coordinate system is usually taken at the midpoint of the bond AB (see Figure 3), and its transformation under rotations is *identical* with the transformations given for p orbitals. In fact, by the binomial expansion formula,[11] we obtain



(a) (b) $\gamma = 0°$

(c) $\beta = -\pi/2 = 3\pi/2$ (d) $\alpha = -\pi/2 = 3\pi/2$

Figure 2.—An example. (a) Original combination of cartesian coordinate systems. (b) Rotation about Z. (c) Rotation about Y'. (d) Rotation about Z''.

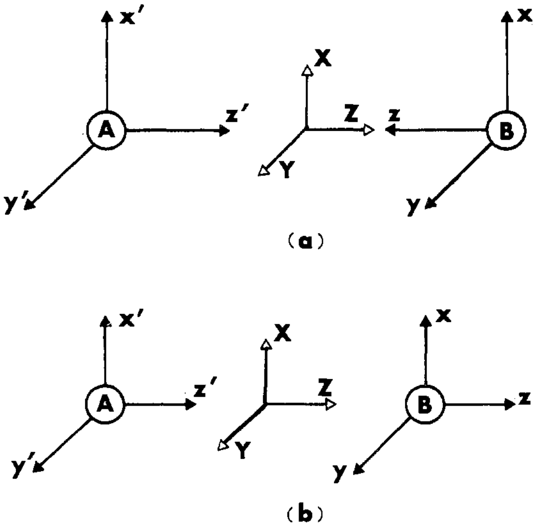


Figure 3.—"Proper" alignment: (a) for oppositely-handed coordinate systems; (b) for right-handed coordinate systems.

$$X^{\alpha} = \sum_{i=0}^{\alpha}\sum_{j=0}^{i} \frac{\alpha!}{(\alpha - i)!j!(i - j)!}[\xi^2 \cos(\alpha + \gamma) - \eta^2 \cos(\alpha - \gamma)]^{\alpha - i}[\xi^2 \sin(\alpha + \gamma) - \eta^2 \sin(\alpha - \gamma)]^{i-j}[2\xi\eta \cos \gamma]^j X_0{}^{\alpha - i} Y_0{}^{i-j} Z_{0_j}$$

$$Y^{\beta} = \sum_{s=0}^{\beta}\sum_{t=0}^{s} \frac{\beta!}{(\beta - s)!t!(s - t)!}[-\xi^2 \sin(\alpha + \gamma) - \eta^2 \sin(\alpha + \gamma)]^{\beta - s}[\xi^2 \cos(\alpha + \gamma) + \eta^2 \cos(\alpha - \gamma)]^{s-t}[-2\xi\eta \sin \gamma]^t X_0{}^{\beta - s} Y_0{}^{s-t} Z_{0_t}$$

---

(10) As a matter of fact overlap integrals are moment integrals in which $\alpha = \beta = \gamma = 0$.

(11) H. Margenau and G. M. Murphy, "The Mathematics of Physics and Chemistry," D. Van Nostrand Co., Inc., New York, N. Y., 1956.

and

$$Z^{\gamma} = \sum_{u=0}^{\gamma}\sum_{v=0}^{u} \frac{\gamma!}{(\gamma - u)!v!(u - v)!}[-2\xi\eta \cos \alpha]^{\gamma-u} \times [-2\xi\eta \sin \alpha]^{u-v}[\xi^2 - \eta^2]^v X_0^{\gamma-u} Y_0^{u-v} Z_0^{v}$$

It is perhaps worth mentioning that a FORTRAN program to evaluate one- and two-center moment integrals of the type

$$\langle \psi_{\mathrm{I}} | X^{\alpha} Y^{\beta} Z^{\gamma} | \psi_{\mathrm{II}} \rangle$$

has been coded by the author and can be obtained from the Quantum Chemistry Program Exchange of Indiana University.

CONTRIBUTION FROM THE METCALF CHEMICAL LABORATORIES
OF BROWN UNIVERSITY, PROVIDENCE, RHODE ISLAND 02912

The Nucleophilic Reactivity of Chlorite Ion as Indicated by Its Reaction Rate with Peroxymonosulfuric Acid[1]

BY ROBERT W. JOHNSON AND JOHN O. EDWARDS

Received June 16, 1966

In a survey of nucleophilic substitution reactions, Edwards and Pearson[2] have shown that the reactivities of nucleophiles can be related to three properties: basicity, polarizability, and an effect not completely understood called the "α effect." Basicity of the nucleophile is the important property when the substrate atom is like a proton, *i.e.*, when it has a localized positive charge and few valence electrons. Polarizability predominates when the substrate atom is electronegative and has a number of outer-orbital electrons. Some nucleophiles are more reactive than expected from their polarizability and basicity. These electron donors, which are said to exhibit the "α effect," are characterized by the presence of one or more unshared pairs of electrons on an electronegative atom adjacent to the nucleophile atom.

The chlorite ion, ClO_2^-, with unshared electrons on the chlorine and oxygen atoms, might be expected to exhibit the "α effect" when acting as a nucleophile and in fact there has been some evidence of high reactivity. It is an intermediate in the two-step disproportionation of hypochlorite

$$ClO^- + ClO^- \longrightarrow ClO_2^- + Cl^-$$
$$ClO^- + ClO_2^- \longrightarrow ClO_3^- + Cl^-$$

The reaction between ClO_2^- and ClO^- is 100 times faster than the first step in the disproportionation.[3] Owing to a common dependence on donation of electrons, nucleophilicity and ability to form complexes often follow parallel orders.[4,5] Gordon and Kern observed that ClO_2^- forms a complex with uranyl ion.[6] The present study was begun to obtain quantitative data on the nucleophilic reactivity of ClO_2^-.

Experimental Section

Reagents.—Laboratory distilled water and reagent grade chemicals were used in all cases. Peroxymonosulfuric acid (Caro's acid) was prepared using Becco 98% H_2O_2 and practical grade HSO_3Cl. The procedure was that of Fortnum.[7] Peroxyacetic acid solutions were prepared by diluting Becco 40% peroxyacetic acid with appropriate buffers.

Analytical Methods.—The peroxy acids were analyzed in the presence of hydrogen peroxide by the method of Greenspan and MacKellar.[8] A 10.0-ml sample of the peroxy acid solution was placed in 150 ml of 5% H_2SO_4 cooled to 0°. Ceric sulfate with ferroin indicator was used to analyze for H_2O_2. Then 5 ml of 20% KI was added and the liberated iodine was titrated with thiosulfate and starch indicator to determine the peroxy acid concentration.

Stoichiometry.—A paper chromatography method[9] was used to analyze for the various species of chlorine. Strips (3 × 20 cm) of Whatman No. 1 filter paper were used. Development time was approximately 10 hr in a 15:2:2:2 mixture of 2-propanol, water, pyridine, and concentrated ammonium hydroxide. Chloride was revealed as a purple spot at R_f 0.25 by spraying with 0.2 N aqueous $AgNO_3$, drying, and exposing to ultraviolet light for 2 min. When the strip was sprayed with equal parts of a 1:1 mixture of 3 M hydrochloric acid–acetone and 5% ethanolic diphenylamine, ClO_2^- was immediately revealed as a blue-green spot at R_f 0.36. After drying, chlorate appeared as a yellow spot at R_f 0.54. (Hypochlorite ion could not be detected in the presence of peroxy acids because both ClO^- and the peroxy acids oxidize I^- with similar rates.) To facilitate comparison of relative concentration of the oxy anion species, solutions of known initial concentrations were applied to the paper using calibrated capillary tubes.

Kinetics.—The peroxy acid was diluted with cold water and KH_2PO_4 buffer was added. Owing to the acid impurity, 1.0 M NaOH was added to bring the pH within the 4.5–7.0 range. The chlorite solution was prepared by dissolving the appropriate weight of $NaClO_2$ in water. The reaction was followed using a Beckman DK-1 spectrophotometer at 260 mμ, the absorption peak of chlorite (ϵ_{260} 176).

Results

Stoichiometry.—The observed stoichiometry was found to be

$$HSO_5^- + ClO_2^- \longrightarrow ClO_3^- + SO_4^{2-} + H^+$$

The pH was kept above 4.6 to minimize decomposition of ClO_2^- and below 7.0 to minimize decomposition of HSO_5^-. Within this pH range and when peroxide was in excess, the absorbance at "infinite time" was that expected for loss of nearly all ClO_2^- and a small fraction of HSO_5^-. The chromatography results showed that the ClO_2^- concentration decreased as the ClO_3^- concentration increased. Also the Cl^- spot was absent or barely detectable. No evidence for ClO^- was found in the ultraviolet spectra.

In the chosen pH range, Caro's acid is present as the mononegative anion[10] and ClO_2^- is the predominant form of the reductant.

---

[1] Taken from the Sc.B. Thesis of R. W. J. at Brown University, 1966.

[2] J. O. Edwards and R. G. Pearson, *J. Am. Chem. Soc.*, **84**, 16 (1962).

[3] M. W. Lister, *Can. J. Chem.*, **34**, 465 (1956).

[4] J. O. Edwards, *J. Am. Chem. Soc.*, **76**, 1540 (1954).

[5] R. G. Pearson, *ibid.*, **85**, 3533 (1963).

[6] G. Gordon and D. M. H. Kern, *Inorg. Chem.*, **3**, 1055 (1964).

[7] D. Fortnum, Ph.D. Thesis, Brown University, 1958, p 47.

[8] F. P. Greenspan and D. G. MacKellar, *Anal. Chem.*, **20**, 1061 (1948).

[9] B. L. Harrison and D. H. Rosenblatt, *J. Chromatog.*, **13**, 271 (1964).

[10] D. L. Ball and J. O. Edwards, *J. Am. Chem. Soc.*, **78**, 1125 (1956).