1982/1983 of G.M. at the Max-Planck-Institut für Kohlenforschung, Mülheim a.d. Ruhr. We are grateful to Mr. K.-H. Claus for the data collection and Prof. Dr. Carl Krüger for the use of the programs. Mr. J. Riede is thanked for the data collection of 3 and Dr. F. R. Kreissl for mass spectroscopic measurements.

Registry No. 1, 64065-06-1; 2, 93646-00-5; 3, 93646-01-6; 4, 93646-02-7; 5, 93646-03-8; 7, 93646-04-9; $AlCl_3$, 7446-70-0; $AlMe_3$, 75-24-1; $AlMe_2Cl$, 1184-58-3.

Supplementary Material Available: Tables of additional crystal structure data, thermal parameters, H atom coordinates, and observed and calculated structure factor amplitudes for 3 and 7 (23 pages). Ordering information is given on any current masthead page.

Reaction of the Phenoxide Ion with Trimethylaluminum. Isolation and Crystal Structure of [K·dibenzo-18-crown-6][Al_2Me_6OPh] and K[$AlMe_2(OPh)_2$]

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Potassium phenoxide reacts cleanly with trimethylaluminum in toluene in the presence of a 1:1 ratio of crown ether to salt. The product [K·dibenzo-18-crown-6][Al_2Me_6OPh], 1, was crystallized by cooling from 70 °C to room temperature. In a 1:2 ratio of crown ether to salt, the temperature must be raised to ca. 160 °C for complete reaction. Slow cooling afforded K[$AlMe_2(OPh)_2$], 2, as the possible decomposition product of $[Al_2Me_6OPh]^-$. 1 belongs to the monoclinic space group $P2_1/n$ with a = 19.763 (6) Å, b = 9.830 (3) Å, c = 20.661 (6) Å, β = 116.00 (2)°, and ρ_{calcd} = 1.18 g/cm^3 for Z = 4. The structure was refined to R = 0.088 with 1756 observed reflections. 2 crystallizes in the orthorhombic space group $Pbcn$ with a = 20.487 (6) Å, b = 10.129 (4) Å, c = 7.259 (3) Å, and ρ_{calcd} = 1.24 g/cm^3 for Z = 4. The refinement based on 483 observed reflections led to R = 0.031. The anion of 1 has a Y-shaped structure with Al–O–Al = 128.3 (7)° and Al–O = 1.86 (1) Å. The plane of the phenyl ring makes a 77° angle with the Al–O–Al plane. The anion of 2 resides on a crystallographic twofold axis with the Al–O length a short 1.800 (3) Å.

Introduction

Our recent investigations into the formation and subsequent decomposition behavior of high oxygen content organoaluminum compounds have resulted in isolation of novel anionic species that are based upon four-[1] or six-membered[2] ring systems. These rings consist of alternating Al–O linkages and the anions so formed must represent intermediates to later stages of decomposition of anions of the type $[Al_2Me_6X]^-$ where X is typically oxide,[2] superoxide,[2,3] or high oxygen content anions such as cacodylate,[1,6] sulfate,[4] nitrate,[5] or acetate.[6] In an effort to further elucidate the pathways through which such decomposition may occur and to extend the range of liquid clathrate forming compounds,[7] we have conducted an investigation into the ability of the phenoxide anion to form liquid clathrates in the presence of trimethylaluminum and aromatic liquids. In this contribution we report structural characterization of both the 2:1 adduct of trimethylaluminum with phenoxide and a second compound that may be the immediate decomposition product of the adduct.

Results and Discussion

Potassium phenoxide reacts rapidly in the presence of dibenzo-18-crown-6 (1 equiv), trimethylaluminum (2 equiv), and toluene (excess) to yield a liquid clathrate of the composition [K·dibenzo-18-crown-6][Al_2Me_6OPh]·nC_6H_5Me. Heating of this system to 70 °C followed by slow cooling to room temperature yielded crystals of [K·dibenzo-18-crown-6][Al_2Me_6OPh], 1, that were characterized crystallographically. However, the presence of only 0.5 equiv of crown ether, yielded both 1 and its decomposition product K[$AlMe_2(OPh)_2$], 2, in the liquid clathrate layer. The presence of 2 was confirmed by the observation of two major resonances at ca. 0 ppm in the ^1H NMR spectrum of the liquid clathrate layer and by the subsequent characterization of 2 through a single-crystal X-ray diffraction study. 2 may represent the initial product in the decomposition pathway of 1, which presumably halts at this early stage (unlike compounds in some of our earlier decomposition studies) because of the relative strength of the O–C bond in the phenoxide anion.

A view of the anion of 1 is given in Figure 1, and it clearly demonstrates adherence to the type of geometry that is typically seen for liquid clathrate parent anions. Similar Y-shaped structures were characterized in the analogous anions derived from azide,[8] superoxide,[3] and chloride.[9] The conformation within the anion is consistent

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(1) Zaworotko, M. J.; Atwood, J. L. *J. Chem. Soc., Chem. Commun.* **1983**, 302.

(2) Atwood, J. L.; Hrncir, D. C.; Priester, R. D.; Rogers, R. D. *Organometallics* **1983**, *2*, 985.

(3) Hrncir, D. C.; Rogers, R. D.; Atwood, J. L. *J. Am. Chem. Soc.* **1981**, *103*, 4277.

(4) Rogers, R. D.; Atwood, J. L. *Organometallics* **1984**, *3*, 271.

(5) Atwood, J. L.; Crissinger, K. D.; Rogers, R. D. *J. Organomet. Chem.* **1978**, *155*, 1.

(6) Zaworotko, M. J.; Rogers, R. D.; Atwood, J. L. *Organometallics* **1982**, *1*, 1179.

(7) For reviews of liquid clathrate behavior see: Atwood, J. L. *Recent Dev. Sep. Sci.*, 1977, *3*, 195. Atwood, J. L. In "Inclusion Compounds"; Atwood, Davies, MacNicol, Eds.; Academic Press: London, 1984; Vol I.

(8) Atwood, J. L.; Newberry, W. R. *J. Organomet. Chem.* **1974**, *65*, 145.

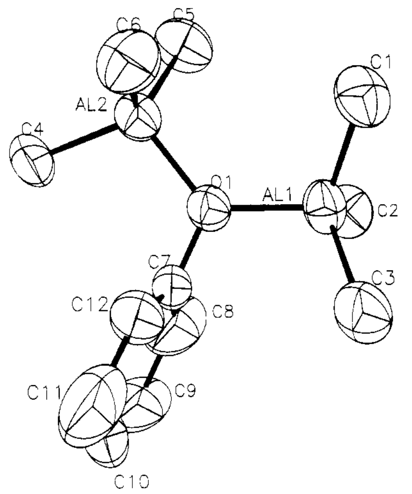

Figure 1. Structure of the $[Al_2Me_6OPh]^-$ anion of 1.

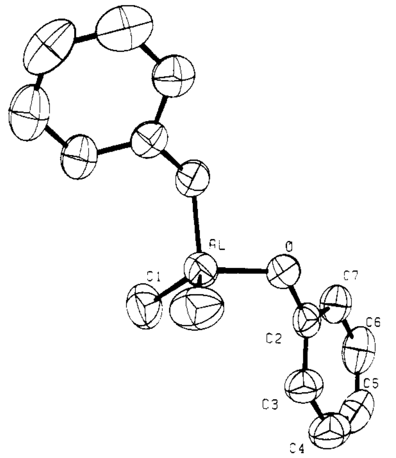

Figure 2. Structure of the $[AlMe_2(OPh)_2]^-$ anion of 2. The aluminum atom resides on a crystallographic twofold axis.

with minimization of steric repulsions, with the methyl groups being staggered and the phenyl ring disposing itself in a roughly perpendicular fashion to the Al–O–Al plane (77°). The absence of strong interactions with the cation prevents influence upon this conformation, unlike the effects arising from interionic attractions that were seen in $Rb[Ga_2Me_6(MeCOO)]$.[6] Strong overall similarities between the anion of 1 and the $[Al_2Me_6O_2]^-$ anion are seen in the Al–O distances (1.88 (1), 1.901 (9) Å and 1.85 (1), and 1.87 (1) Å, respectively) and the Al–O–Al angles (127.5 (6) and 128.3 (7)°, respectively). The Al–C(methyl) distances, averaging 2.01 (2) Å, and the C–Al–C angles are unexceptional. The oxygen atom of the anion and the three atoms bound to it are planar to within 0.008 Å. This supports the concept of sp^2 hybridization of the oxygen atom.

The potassium of the cation lies almost symmetrically within the crown ether such that K–O distances range from 2.72 (1) to 2.79 (1) Å. The potassium cation lies 0.39 Å out of the plane formed by the six crown ether oxygen atoms. This geometry is consistent with that seen for the analagous cation in [K·dibenzo-18-crown-6]$[Al_2Me_6Cl]$.[9]

The view of 2, which is presented in Figure 2, reveals that decomposition of 1 has resulted in the formation of a product that contains an $AlMe_2$ bridging unit. This bridging moiety not only is characteristic of some of our aforementioned studies but also has precedent in studies conducted upon the reactivity of trimethylaluminum with amines.[10-13] The anion resides on a crystallographic

Table I. Crystal Data and Summary of Intensity Data Collection and Structure Refinement

	[K·dibenzo-18-crown-6]-$[Al_2Me_6OPh]$	$K[AlMe_2(OPh)_2]$
mol wt	636.8	282.3
cell dimens		
a, Å	19.763 (6)	20.487 (6)
b, Å	9.830 (3)	10.129 (4)
c, Å	20.661 (6)	7.259 (3)
β, deg	116.00 (2)	
space group	$P2_1/n$	*Pbcn*
cell vol, Å^3	3592.2	1506.3
molecules/unit cell	4	4
ρ(calcd), g cm^{-3}	1.18	1.24
μ(calcd), cm^{-1}	2.4	4.0
radiatn	Mo Kα	Mo Kα
max cryst dimens, mm	0.15 × 0.20 × 0.30	0.20 × 0.30 × 0.35
scan width	0.80 + 0.20 tan *θ*	0.80 + 0.20 tan *θ*
variatn of stds	±2%	±2%
reflctns obsd	1756	483
2*θ* range	4–50	4–36
no. of parameters varied	379	83
GOF	0.90	1.0
R	0.088	0.031
R_w	0.093	0.031

Table II. Final Fractional Coordinates for [K·dibenzo-18-crown-6]$[Al_2Me_6OPh]$, 1

atom	*x/a*	*y/b*	*z/c*
K	0.0784 (2)	0.7119 (4)	0.3444 (2)
Al(1)	0.3282 (2)	0.5232 (5)	0.6552 (2)
Al(2)	0.2647 (3)	0.4543 (5)	0.4773 (2)
O(1)	0.3229 (5)	0.552 (1)	0.5621 (4)
C(1)	0.3050 (9)	0.326 (1)	0.6586 (8)
C(2)	0.2518 (9)	0.644 (2)	0.6637 (8)
C(3)	0.4356 (8)	0.570 (2)	0.7225 (9)
C(4)	0.2608 (9)	0.568 (2)	0.3950 (8)
C(5)	0.1596 (8)	0.432 (2)	0.4724 (9)
C(6)	0.3210 (9)	0.280 (2)	0.4848 (9)
C(7)	0.366 (1)	0.663 (3)	0.5558 (8)
C(8)	0.340 (1)	0.796 (3)	0.556 (1)
C(9)	0.392 (2)	0.900 (3)	0.550 (1)
C(10)	0.459 (3)	0.848 (7)	0.550 (2)
C(11)	0.480 (3)	0.728 (5)	0.544 (3)
C(12)	0.433 (1)	0.617 (3)	0.553 (1)
OC(1)	0.1586 (5)	0.913 (1)	0.3116 (6)
OC(2)	0.1073 (5)	0.679 (1)	0.2259 (5)
OC(3)	0.0130 (7)	0.516 (1)	0.2436 (6)
OC(4)	−0.0463 (6)	0.573 (1)	0.3403 (7)
OC(5)	0.0203 (7)	0.785 (1)	0.4390 (7)
OC(6)	0.1186 (6)	0.947 (1)	0.4252 (6)
CC(1)	0.1400 (9)	0.911 (2)	0.2337 (8)
CC(2)	0.1617 (8)	0.773 (2)	0.2182 (9)
CC(3)	0.106 (1)	0.549 (2)	0.2027 (9)
CC(4)	0.055 (1)	0.457 (2)	0.2101 (9)
CC(5)	0.047 (1)	0.323 (2)	0.186 (1)
CC(6)	0.094 (2)	0.282 (2)	0.151 (1)
CC(7)	0.146 (1)	0.374 (3)	0.146 (1)
CC(8)	0.1506 (9)	0.506 (2)	0.1678 (9)
CC(9)	−0.047 (1)	0.428 (2)	0.246 (1)
CC(10)	−0.093 (1)	0.524 (2)	0.267 (1)
CC(11)	−0.090 (1)	0.658 (2)	0.367 (1)
CC(12)	−0.040 (1)	0.696 (2)	0.441 (1)
CC(13)	0.067 (1)	0.856 (2)	0.501 (1)
CC(14)	0.121 (1)	0.942 (2)	0.493 (1)
CC(15)	0.171 (1)	1.017 (2)	0.552 (1)
CC(16)	0.163 (2)	1.004 (3)	0.617 (1)
CC(17)	0.113 (2)	0.919 (3)	0.624 (2)
CC(18)	0.063 (1)	0.845 (2)	0.566 (2)
CC(19)	0.1703 (9)	1.041 (2)	0.414 (1)
CC(20)	0.1448 (9)	1.046 (2)	0.3323 (9)

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[9] Atwood, J. L.; Hrncir, D. C.; Rogers, R. D. *J. Inclusion Phenom.* **1983**, *1*, 199.

Table III. Bond Lengths (Å) and Angles (deg) for 1

	Bond Distances		
K-Oc(1)	2.79 (1)	K-Oc(2)	2.77 (1)
K-Oc(3)	2.72 (1)	K-Oc(4)	2.78 (1)
K-Oc(5)	2.76 (1)	K-Oc(6)	2.75 (1)
Al(1)-O(1)	1.901 (9)	Al(1)-C(1)	2.00 (2)
Al(1)-C(2)	1.98 (1)	Al(1)-C(3)	2.01 (2)
Al(2)-O(1)	1.88 (1)	Al(2)-C(4)	2.01 (2)
Al(2)-C(5)	2.04 (1)	Al(2)-C(6)	2.01 (2)
O(1)-C(7)	1.43 (2)	C(7)-C(8)	1.40 (2)
C(7)-C(12)	1.42 (3)	C(8)-C(9)	1.48 (3)
C(9)-C(10)	1.41 (5)	C(10)-C(11)	1.27 (8)
C(11)-C(12)	1.49 (5)		
	Bond Angles		
O(1)-Al(1)-C(1)	105.4 (6)	O(1)-Al(1)-C(2)	107.1 (5)
C(1)-Al(1)-C(2)	112.8 (7)	O(1)-Al(1)-C(3)	103.9 (6)
C(1)-Al(1)-C(3)	112.7 (7)	C(2)-Al(1)-C(3)	114.1 (7)
O(1)-Al(2)-C(4)	106.7 (6)	O(1)-Al(2)-C(5)	106.9 (6)
C(4)-Al(2)-C(5)	110.8 (7)	O(1)-Al(2)-C(6)	105.8 (6)
C(4)-Al(2)-C(6)	110.8 (7)	C(5)-Al(2)-C(6)	115.2 (7)
Al(1)-O(1)-Al(2)	127.5 (6)	Al(1)-O(1)-C(7)	115.8 (8)
Al(2)-O(1)-C(7)	116.7 (8)	O(1)-C(7)-C(8)	119 (2)
O(1)-C(7)-C(12)	111 (2)	C(8)-C(7)-C(12)	130 (2)
C(7)-C(8)-C(9)	112 (2)	C(8)-C(9)-C(10)	115 (3)
C(9)-C(10)-C(11)	133 (6)	C(10)-C(11)-C(12)	115 (6)
C(7)-C(12)-C(11)	114 (3)		

twofold axis. The Al–O distances are 1.800 (3) Å and therefore lie on the short side of the range typically observed for such linklages.[14] The angles around the aluminum atom reflect the fact that methyl groups are more sterically cumbersome than phenoxy groups. The O–Al–O angle is acute (96.8 (2)°), the C–Al–C angle is obtuse (119.4 (3)°), and the O–Al–C angles are closest to the classic tetrahedral value (108.7 (2)°). The steric crowding around aluminum also manifests itself in the somewhat wide Al–O–C angles of 125.0 (3)°. This feature may also, of course, be attributable to sp^2 character in the oxygen atom. M–O–C angles seen in other metal phenoxides where steric crowding prevails are similar, as evidenced by the OAr (OAr = 2,6-di-*tert*-butyl-4-methylphenoxide) complexes $M(OAr)_2$[15] (M = Ge, Sn, Pb) and $(C_5H_5)_2TiOAr$.[16]

The cation environment is similar to that previously found in $Rb[Ga_2Me_6(MeCOO)]$.[6]

In conclusion, we have been able to isolate another probable decomposition product of an oxygen-containing trimethylaluminum adduct. Further investigations into such behavior are under way.

Experimental Section

All reactions were carried out under a nitrogen atmosphere by using Schlenk techniques or an inert-atmosphere glovebox. Solvents were dried and degassed in the normal manner. Trimethylaluminum was purchased from Alfa Products, and the crown ether was purchased from Aldrich Chemical Co., both being used without further purification. Potassium phenoxide was prepared according to the literature method.[18]

Table IV. Final Fractional Coordinates for $K[AlMe_2(OPh)_2]$, 2

atom	*x/a*	*y/b*	*z/c*
K	0.0000	−0.1060 (1)	0.7500
Al	0.0000	0.2471 (2)	0.7500
O	−0.0464 (1)	0.1291 (3)	0.8814 (4)
C(1)	−0.0601 (2)	0.3451 (5)	0.5884 (7)
C(2)	−0.1067 (2)	0.1512 (5)	0.9531 (6)
C(3)	−0.1192 (2)	0.2626 (5)	1.0580 (7)
C(4)	−0.1792 (3)	0.2802 (6)	1.1378 (7)
C(5)	−0.2286 (3)	0.1899 (7)	1.1134 (8)
C(6)	−0.2164 (3)	0.0784 (7)	1.0100 (8)
C(7)	−0.1554 (3)	0.0573 (5)	0.9301 (6)

Preparation of [K·dibenzo-18-crown-6][Al_2Me_6OPh], 1. Twenty milliliters of toluene were added to 5 mmol of KOPh and 5 mmol of dibenzo-18-crown-6. Upon addition of 10 mmol of $AlMe_3$, reaction occurred immediately. After 20 h at room temperature all solid had dissolved and a brownish liquid clathrate layer was evident. Heating to 70 °C followed by slow cooling to room temperature afforded clear, colorless, extremely air-sensitive crystals of 1.

Preparation of $K[AlMe_2(OPh)_2]$, 2. Using the procedure outlined for 1, but with only 2.5 mmol of dibenzo-18-crown-6, a brown liquid clathrate was obtained. The closed reaction flask was heated in a sand bath at 160 °C for 24 h, after which all solid had been consumed. Slow cooling afforded crystals of 2 that were suitable for X-ray diffraction analysis.

X-ray Data Collection, Structure Determination, and Refinement for 1. An air- and moisture-sensitive crystal of 1 was sealed in a capillary in an inert-atmosphere glovebox and placed on an Enraf-Nonius CAD-4 diffractometer. Final lattice parameters as determined from a least-squares refinement of $((\sin\theta)/\lambda)^2$ values for 15 reflections ($\theta > 15°$) accurately centered on the diffractometer are given in Table I.

Data were collected by the previously described method,[19] and a summary of data collection parameters is given in Table I. The structure was solved through application of the MULTAN[20] program and all subsequent calculations were carried out with the SHELX[21] system of computer programs. Neutral atom scattering factors for K, Al, O, and C were taken from Cromer and Waber,[22]

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(10) McLaughlin, G. M.; Sim, G. A.; Smith, J. D. *J. Chem. Soc., Dalton Trans.* **1972**, 2197.

(11) Alford, K. J.; Gosling, K.; Smith, J. D. *J. Chem. Soc., Dalton Trans.* **1972**, 2203.

(12) Beachley, O. T.; Racette, K. C. *Inorg. Chem.* **1975**, *14*, 2534.

(13) Beachley, O. T.; Racette, K. C. *Inorg. Chem.* **1976**, *15*, 2110.

(14) See Table V in ref 6.

(15) Cetinkaya, B.; Gumrukcu, I.; Lappert, M. F.; Atwood, J. L.; Rogers, R. D.; Zaworotko, M. J. *J. Am. Chem. Soc.* **1980**, *102*, 2088.

(16) Cetinkaya, B.; Hitchcock, P. B.; Lappert, M. F.; Torroni, S.; Atowod, J. L.; Hunter, W. E.; Zaworotko, M. J. *J. Organomet. Chem.* **1980**, *188*, C31.

(17) Pauling values obtained from: Cotton, F. A.; Wilkinson, G. "Advanced Inorganic Chemistry", 4th Ed.; Wiley-Interscience: New York, 1980.

(18) Kornblum, N.; Lurie, A. P. *J. Am. Chem. Soc.* **1959**, *81*, 2707.

(19) Holton, J.; Lappert, M. F.; Ballard, D. G. H.; Pearce, R.; Atwood, J. L.; Hunter, W. E. *J. Chem. Soc., Dalton Trans.* 1979, 46.

(20) Germain, G.; Main, P.; Woolfson, M. M. *Acta Crystallogr., Sect. A* 1971, *A27*, 368.

(21) SHELX, a system of computer programs for X-ray structure determination by G. M. Sheldrick, 1976. Other programs used included ORTEP by C. K. Johnson and BPL by W. E. Hunter.

Table V. Bond Lengths (Å) and Angles (deg) for 2

	Bond Distances		
Al-O	1.800 (3)	Al-C(1)	1.968 (5)
O-C(2)	1.359 (5)	C(2)-C(3)	1.384 (6)
C(2)-C(7)	1.389 (6)	C(3)-C(4)	1.372 (6)
C(4)-C(5)	1.376 (7)	C(5)-C(6)	1.379 (7)
C(6)-C(7)	1.394 (6)		
	Bond Angles		
O-Al-C(1)	108.7 (2)	Al-O-C(2)	125.0 (3)
O-Al-O	96.8 (2)	C(1)-Al-C(1)	119.4 (3)
O-C(2)-C(3)	120.9 (4)	O-C(2)-C(7)	119.6 (4)
C(3)-C(2)-C(7)	119.5 (4)	C(2)-C(3)-C(4)	120.3 (5)
C(3)-C(4)-C(5)	121.2 (5)	C(4)-C(5)-C(6)	118.8 (6)
C(5)-C(6)-C(7)	121.0 (5)	C(2)-C(7)-C(6)	119.2 (5)

and the anomalous dispersion corrections applied were those of Cromer and Liberman.[23]

Full-matrix least-squares refinement of all non-hydrogen atoms using anisotropic thermal parameters led to final values of $R = \sum||F_o| - |F_c||/\sum|F_o| = 0.088$ and $R_w = [\sum w(F_o - F_c)^2/\sum w(F_o)^2]^{1/2} = 0.093$. No hydrogen atoms were locatable on the final difference Fourier map, and no correction for absorption was applied (μ = 2.5 cm^{-1}). The final values of the fractional coordinates are listed in Table II, and thermal parameters are supplied as supplementary material.[24] Bond distances and angles are given in Table III.

X-ray Data Collection, Structure Determination, and Refinement for 2. Data collection procedures were the same as for 1, and pertinent parameters are given alongside those for 1 in Table I. The structure was solved via MULTAN and refined by using the SHELX program package. The aluminum and potassium atoms were shown to lie on twofold positions in the space group *Pbcn*. With all non-hydrogen atoms refined with anisotropic thermal parameters and hydrogen atoms fixed in positions located from a difference Fourier, final values of R = 0.031 and R_w = 0.031 were obtained. No correction for absorption was applied (μ = 4.0 cm^{-1}). Final fractional coordinates are given in Table IV, and bond distances and angles are presented in Table V. Thermal parameters are supplied as supplementary material. Scattering factors for H were taken from ref 25.

Acknowledgment. We are grateful to the National Science Foundation for support of this work.

Registry No. 1, 91711-78-3; 2, 91711-79-4.

Supplementary Material Available: Tables of bond lengths and angles and observed and calculated structure factors for 1 and 2, thermal parameters for 1, and hydrogen atom coordinates for 2 (18 pages). Ordering information is given on any current masthead page.

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(22) Cromer, D. T.; Waber, J. T. *Acta Crystallogr.* **1965**, *18*, 104.
(23) Cromer, D. T.; Libermann, D. M. *J. Chem. Phys.* **1970**, *53*, 1891.
(24) See paragraph at the end of paper regarding supplementary material.
(25) "International tables for X-ray Crystallography"; Kynoch Press: Birmingham, England, 1974; Vol. IV, p 72.

Synthesis and Crystal and Molecular Structure of an η-Cyclopropene Complex of Molybdenum

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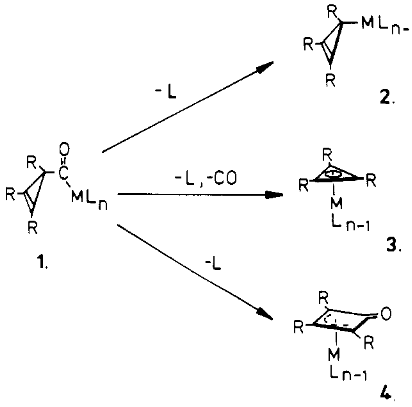
The 16-electron cation $[Mo(C_5Me_5)(CO)_3]^+$ (6) reacts with sodium 2,3-diphenyl-2-cyclopropene-1-carboxylate with displacement of CO and formation of the chelate complex 9, containing the first example of a molybdenum–cyclopropene bond. Complex 9 has been completely characterized spectroscopically and by a single-crystal X-ray diffraction study. Crystals of 9 are monoclinic of space group $P2_1/c$ (No. 14, C_{2h}^5) with a = 16.638 (5) Å, b = 11.526 (3) Å, c = 15.892 (5) Å, β = 114.54 (2)°, V = 2772.4 (13) Å^3, Z = 4, R_F = 0.0493, R_{wF} = 0.0509, and GOF = 1.235. ^{13}C NMR studies indicate that the solid-state structure of 9 is maintained in solution.

Introduction

Although the transition-metal chemistry of cyclopropenes has been investigated extensively, simple η^2-cyclopropene complexes are rare and have only been characterized chemically and spectroscopically for niobium[3] and crystallographically for platinum.[4,5] The two most common modes of reactivity involve cyclooligomerization reactions[6,7] and ring-opening reactions to give η-allyl[8,9] or vinylketene[10-12] complexes. We have been particularly interested in the chemistry of (2-cyclopropene-1-carbonyl)transition metal compounds[13] 1 and

Scheme I



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(3) Fredericks, S.; Thomas, J. L. *J. Am. Chem. Soc.* **1978**, *100*, 350–351.
(4) Visser, J. P.; Schipperijn, A. J.; Lukas, *J. Organomet. Chem.* **1973**, *47*, 433–438.
(5) DeBoer, J. J.; Bright, D. *J. Chem. Soc., Dalton Trans.* **1975**, 662–665.
(6) Binger, P.; Doyle, M. J.; McMeeking, J.; Kruger, C. *J. Organomet. Chem.* **1977**, *135*, 405–414.
(7) Binger, P.; Doyle, M. J. *J. Organomet. Chem.* **1978**, *162*, 195–207.
(8) Fiato, R.; Mushak, P.; Battiste, M. A. *J. Chem. Soc., Chem. Commun.* **1975**, 869–871.
(9) Mushak, P.; Battiste, M. A. *J. Organomet. Chem.* **1969**, *17*, P46–P48.
(10) Dettlaf, G.; Behrens, U.; Weiss, E. *Chem. Ber.* **1978**, *111*, 3019–3028.