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Powder Structure Solutions of the Compounds Potassium Phenoxide−Phenol: $C_6H_5OK \cdot xC_6H_5OH$ ($x = 2, 3$)

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We report the ab initio structure solutions of two solvent containing potassium phenoxides by high-resolution powder X-ray diffraction. Both compounds are of interest for the classification of the mechanism of Kolbe−Schmitt type reactions. $C_6H_5OK \cdot 2C_6H_5OH$ crystallizes in space group *Abm*2, $Z = 4$, with unit cell parameters $a = 10.12458(4)$ Å, $b = 21.2413(1)$ Å, $c = 7.89784(3)$ Å. $C_6H_5OK \cdot 3C_6H_5OH$ crystallizes in space group *Pbca*, $Z = 8$, with unit cell parameters $a = 22.7713(1)$ Å, $b = 25.4479(2)$ Å, $c = 7.75549(4)$ Å. Both compounds show polymeric zigzag chains $^{1}_{\infty}[K^{[6]}O_2^{[2]}O^{[1]}\pi(\text{phenyl})^{[1]}]$ aligned along the *c*-axis. The coordination of the potassium ions is similar for both compounds. They lie at the center of distorted octahedra of five oxygen atoms and one phenyl ring, which donates its π electrons. The distortion decreases as the number of free phenol increases.

Introduction

The carboxylation of alkali phenolates, known as Kolbe−Schmitt synthesis,[1] is an industrially important solid−gas reaction. It is the first step in the pathway of producing many pigments, fertilizers and pharmaceuticals. Although the reaction is known since the middle of the last century, its mechanism and even the structures of some of the reactants and products are still a mystery.[2] Many models for the reaction mechanism have been published so far.[3] However, only few studies investigated the influence of solvents on the reactants of the Kolbe−Schmitt synthesis so far.[2,4] Since at least some amount of phenol is present during the process, it is very likely that not only phenoxide but also phenoxide−phenol complexes take part in the carboxylation and influence the type and the amount of the reaction products. For example, in the Wacker process the carboxylation is performed in a melt of phenol.[5] The major problem in studying these structures is that single crystals cannot be grown and powder diffraction data have proved to be too difficult to interpret up to now. Recently, we reported the crystal structure of solvent free C_6H_5OK as determined using high-resolution powder diffraction.[6]

Previous work has shown that the potassium phenoxide − phenol system ($C_6H_5OK \cdot nC_6H_5OH$) contains two members with $n = 2, 3$,[7] but all attempts to grow single crystals have failed. Therefore, powder diffraction techniques are the only means available, and in the present paper, we report the structures of $C_6H_5OK \cdot 2C_6H_5OH$ (**I**) and $C_6H_5OK \cdot 3C_6H_5OH$ (**II**). To determine these structures from powder diffraction patterns, we used the same new "pseudoatom" method which was used for the structure solution of C_6H_5OK.[6] We find a remarkable π interaction between the electron system of one phenyl ring and the metal.

Experimental Section

Materials. All manipulations of solvents and substances were carried out in dry argon using standard Schlenk and vacuum techniques. Pentane was purified and dried according to the standard procedures. Toluene was predried over sodium and freshly distilled from sodium benzophenone ketyle. Phenol was sublimated at a cooling finger at room temperature under reduced pressure (0.1 Torr) and kept in dry argon in a Schlenk vessel. A known amount of potassium cuttings was covered with toluene, heated until melting, stirred rapidly, and quenched without further stirring. A solution of a stoichiometric amount of phenol in toluene was added dropwise over 2 h to the stirred potassium sand at 0 °C. The reaction mixture was allowed to warm to room temperature and stirred for another 2 h. Afterward, toluene was removed from the white precipitate via a filter cannula. The solid was washed free of toluene with pentane and filtered off. The pentane was removed in a vacuum (0.005 Torr) for 1 day at room temperature while the powder was vigorously stirred. The obtained compounds were stored in a Schlenk vessel.

Powder X-ray Diffraction Experiments. For the X-ray powder diffraction experiments, the air and moisture sensitive samples were sealed in glass capillaries of 0.7 mm diameter. High-resolution powder diffraction data were collected at the SUNY X3B1 beamline at the National Synchrotron Light Source, Brookhaven National Laboratory. X-rays of wavelength 1.14939(2) Å for **I** and 1.14853(2) Å for **II** were selected by a double Si(111) monochromator. Wavelengths and the zero point were determined from eight well-defined reflections of the NIST1976 flat plate alumina standard. The diffracted beam was

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Table 1. Lattice Parameters and Selected Details of Refinements of $C_6H_5OK{\cdot}2(C_6H_5OH)$ and $C_6H_5OK{\cdot}3(C_6H_5OH)^a$

	$C_6H_5OK{\cdot}2C_6H_5OH$	$C_6H_5OK{\cdot}3C_6H_5OH$
a [Å]	10.12458(4)	22.7713(1)
b [Å]	21.2413(1)	25.4479(2)
c [Å]	7.89784(3)	7.75549(4)
V [Å^3]	1698.50(1)	4494.18(4)
V/Z [Å^3]	424.63	561.87
Z	4	8
space group	*Abm*2	*Pbca*
calcd density [g/cm^3]	1.245	1.216
R_p [%]	5.09	6.61
R_{wp} [%]	7.36	9.16
R_F [%]	5.94	8.86
no. of obsd reflcns	274	1551
no. of refined atoms (non H)	21 (13)	49 (29)

^{a} R_p, R_{wp}, and R_F refer to the Rietveld criteria of fit for profile, weighted profile, and structure factor, respectively, defined in ref 30. The R factors of **II** are a bit higher than normally accepted due to unidentified impurities.

analyzed with a Ge(111) crystal and detected with a Na(Tl)I scintillation counter with a pulse height discriminator in the counting chain. The incoming beam was monitored by an ion chamber for normalization for the decay of the primary beam. In this parallel beam configuration, the resolution is determined by the analyzer crystal instead of by slits. Data were taken at room temperature for 6.2 s at each 2θ in steps of 0.005° from 4 to 55.2° for **I** and for 5.2 s at each 2θ in steps of 0.005° from 4 to 60.0° for **II**.

Although θ scans did not show serious crystallite size effects, both samples were rocked around θ for 2° during measurement for better statistics. Low angle diffraction peaks had a full width at half-maximum of 0.013° 2θ for **I** and of 0.012° 2θ for **II**, both were not significantly broader than the resolution of the spectrometer. To decrease the thermal motion of the atoms and to evaluate the possibility of a distortion at low temperature, **II** was cooled to 20 K in a closed-cycle helium cryostat. No phase transition was evident, although the peaks broadened continuously when cooling (fwhm of 0.023° 2θ at 20 K) and they sharpen again when heating to room temperature in a completely reversible way. This might be due to an inhibited phase transition at lower temperature.

Each of the two room-temperature diffraction patterns could be indexed on the basis of orthorhombic lattices (Table 1).[8] The possible space groups are *Abm*2 and *Abmm* for **I** and *Pbca* for **II**. The number of formula units per unit cell (Z) directly follows from geometrical considerations. A Le-Bail fit[9] using the program FULLPROF[10] worked well to extract about 270 integrated intensities up to 55° 2θ for **I** and about 900 integrated intensities up to 50° 2θ for **II**. These were used as input for the direct methods program SIRPOW92.[11] As in the case of C_6H_5OK it was possible to detect the positions of potassium and some candidate oxygen atoms, but none of the carbon atoms.[6] All attempts to solve the structure by subsequent Rietveld refinements[12] in combination with difference Fourier analysis failed. This can be understood, since the contribution to the scattering of the heavier atoms is too small.

The pseudoatom method was then applied to solve these structures. The basic idea is that the intensities of low-angle reflections are sensitive to the number and kind of atoms in the unit cell, but that they are not very sensitive to their exact position. In other words, low-angle reflection peaks are sensitive to the center of gravity location of rigid groups of atoms but not to their orientation. It was therefore proposed that it is a reasonable approximation to replace functional groups such as C_6H_5O by a spherical shell of the appropriate scattering power and with appropriate radii. The contribution of this pseudo atom to the structure factor is known, and it depends only on the position of its center. A similar idea is of importance in protein crystallography.[13−15] As a first approximation, the fragment can also be replaced by the form factor of an existing atom, having the same number of electrons as the entire fragment combined with a rather high-temperature factor in order to smear out the electron density over the entire range of the fragment.

Starting with the partial structure of potassium and oxygen atoms, a pseudo atom was introduced for each of the C_6H_5O groups. Using Rietveld refinements against the low angle part of the diffraction pattern ($\sin \theta_{max}/\lambda = 0.22$), the positions of these pseudoatoms were determined in an iterative procedure. Then, the pseudoatoms were replaced by the corresponding C_6H_5O group, one at a time. The correct location of each group was determined by an orientation—and limited translation—search, optimizing the fit to integrated intensities of the low angle part of the powder pattern. A partial Rietveld refinement including the best-oriented rigid body has been performed prior to the next back-substitution. During all of these steps, the difference electron density around the rigid bodies was checked in order to avoid false minima. At the end of the iteration the coordinates for all of the non-hydrogen atoms of both compounds could be determined.

The final Rietveld refinements were performed using the program package GSAS (Figure 1a,b).[16] The peak profile function was modeled using a multiterm Simpson's rule integration of the pseudo-Voigt function.[17] The strong asymmetry in the low-angle region was modeled by a recently implemented function which accounts for the asymmetry due to axial divergence,[18] leading to a strongly improved fit and therefore better profile R factors. A manually fit background[19] was used in combination with a refinable four-term cosine series. To stabilize the refinement, we have been using flexible rigid bodies for the phenyl rings, allowing the refinement of the average C−C and C−O distances, while keeping the hexagonal symmetry of the C_6 ring. It is unnecessary to determine this molecular moiety which has a well-established structure. This important step reduced the number of independent positional parameters of the non-hydrogen atoms of one phenyl ring from 21 down to 8 (3 rotational, 3 translational, and 2 bond lengths). The positions of most of the non-hydrogen atoms remained close to their original positions after refining their positions independently in a final cycle of refinement without improving the statistics of the fit. The R values are listed in Table 1. The coordinates (using rigid bodies) are given in the Supporting Information. A selection of intra- and intermolecular distances is given in Table 2.

Attempts to find a center of symmetry in **I** failed and confirmed *Abm*2 as the correct space group. The position of the hydrogen atoms could not be determined by powder techniques in this case, but their contribution to the profile is definitely measurable.[20] Inclusion of the phenol H atoms at calculated positions reduced the profile R factors typically 0.5% which can be considered as significant.

Traces of additional phases were present in the scans. In the case of **I** the additional phase could be identified as C_6H_5OK.[6] Inclusion of this phase in the refinement process reduced the weighted profile R factor by a factor of 2. The weight fraction of C_6H_5OK refined to 5.5%. In the case of **II**, the regions with the strongest peaks of the

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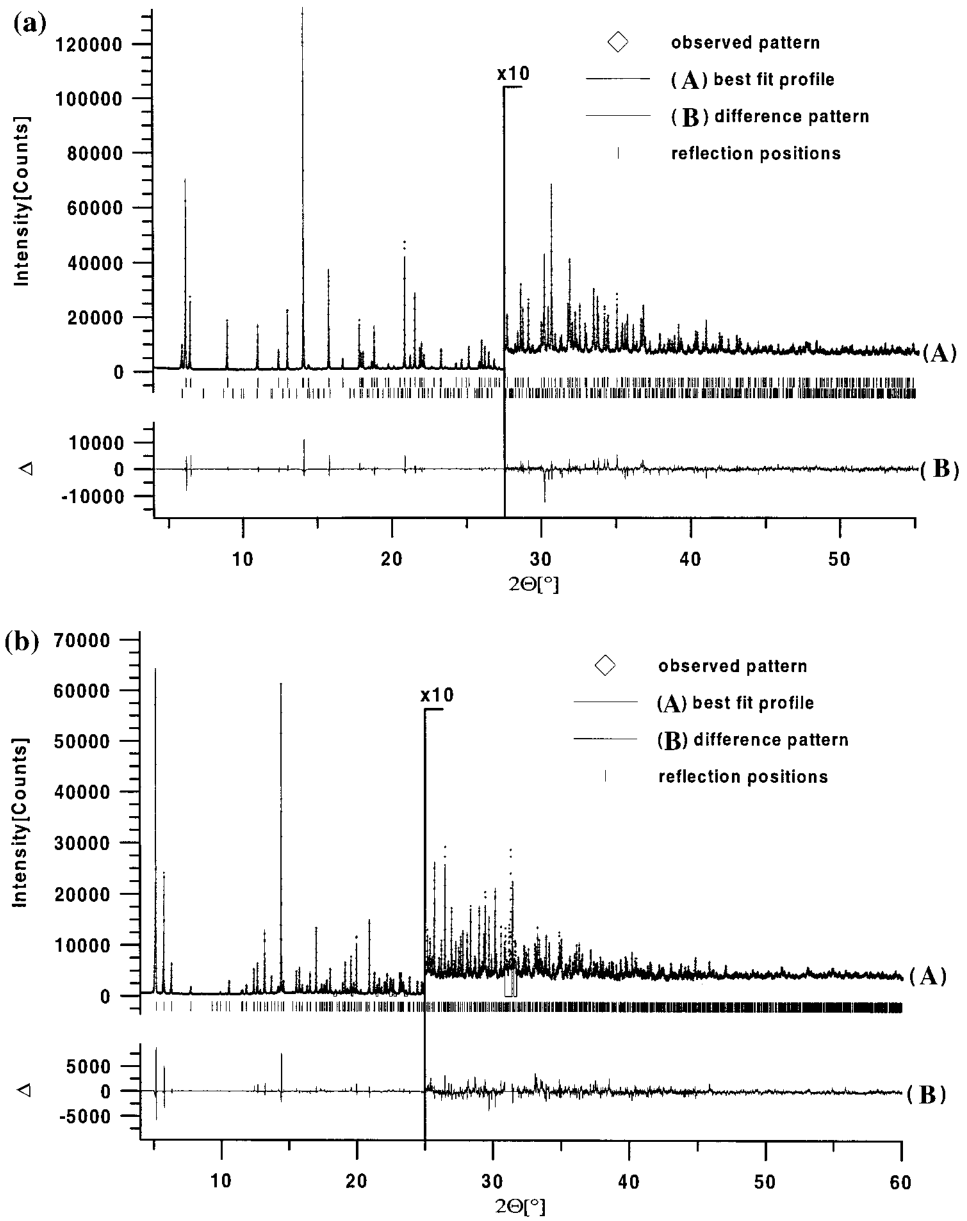

Figure 1. (a) Rietveld plot of orthorhombic $C_6H_5OK{\cdot}2(C_6H_5OH)$. The asymmetric unit contains 21 atoms (13 non-hydrogen atoms). (b) Rietveld plot of orthorhombic $C_6H_5OK{\cdot}3(C_6H_5OH)$. The asymmetric unit contains 49 atoms (29 non-hydrogen atoms).

Table 2. Selected Bond Distance Ranges and Intramolecular Distances of $C_6H_5OK{\cdot}2(C_6H_5OH)$ and $C_6H_5OK{\cdot}3(C_6H_5OH)$ in Å (Esd's Are a Factor of 6 Larger than Rietveld Statistical Estimates, As Discussed in Ref 30)

	$C_6H_5OK{\cdot}2C_6H_5OH$	$C_6H_5OK{\cdot}3C_6H_5OH$
K−K (short)	4.37(1)	4.43(4)
K−O	2.72(2)−2.98(2)	2.84(4)−3.06(4)
K−C (short)	3.20(2)	3.27(3)
K−$\pi(C_6H_5O)$	2.91(2)	3.15(3)
C−O	1.35(3)	1.37(2)
C−C (phenyl)	1.39(1), 1.41(1)[a]	1.40(1)
C−H (phenyl) fixed	1.06	1.05
H−H (shortest interchain)	2.54(3)	2.37(3)
O−O (shortest)	2.41(2)	2.40(2)

[a] The carbon−carbon bond distances of the phenoxide and the phenol have been refined separately for $C_6H_5OK{\cdot}2(C_6H_5OH)$.

unidentified second phase have been excluded from the refinement process.

Description of the Structure. The structure of **I** is based on zigzag chains of edge sharing polyhedra of potassium ions in trans position along the *c* axis (Figure 2a). Every potassium ion is coordinated to five oxygen atoms and one phenyl ring comprising highly distorted octahedra with an K−O−K−O torsion angle of 99.7° along the *c* axis. The phenolate ion is bonded to two potassium ions, one potassium ion is bonded to the oxygen, while the other ion is above the center of the 6-ring, representing a μ^2-bond. The same type of functionality although with different coordination spheres has been found for the phenolate ion and/or the phenol molecule in the stoichiometrical analogous complexes with sodium[21] and cesium.[22] Accordingly, the general formula for the chains is $^{1}_{\infty}[K^{[6]}O_2^{[2]}O^{[1]}\pi{-}\mathrm{phenyl}^{[1]}]$. The strong distortions of the octahedra can be attributed to steric requirements of the bulky phenolate ion. The positions of the two hydroxy hydrogen atoms of the phenols could not be found. Therefore, the distinction between phenol and phenolate ion had to be made according to geometrical considerations. Hydrogen bonding between the phenol oxygen atoms (the two bridging oxygen atoms) and the phenolate oxygen atom are indicated by very short distances (2.4 Å), which also firm the configuration of the chain. The packing density is 68.6%, which is a typical value for this type of organometallic compounds

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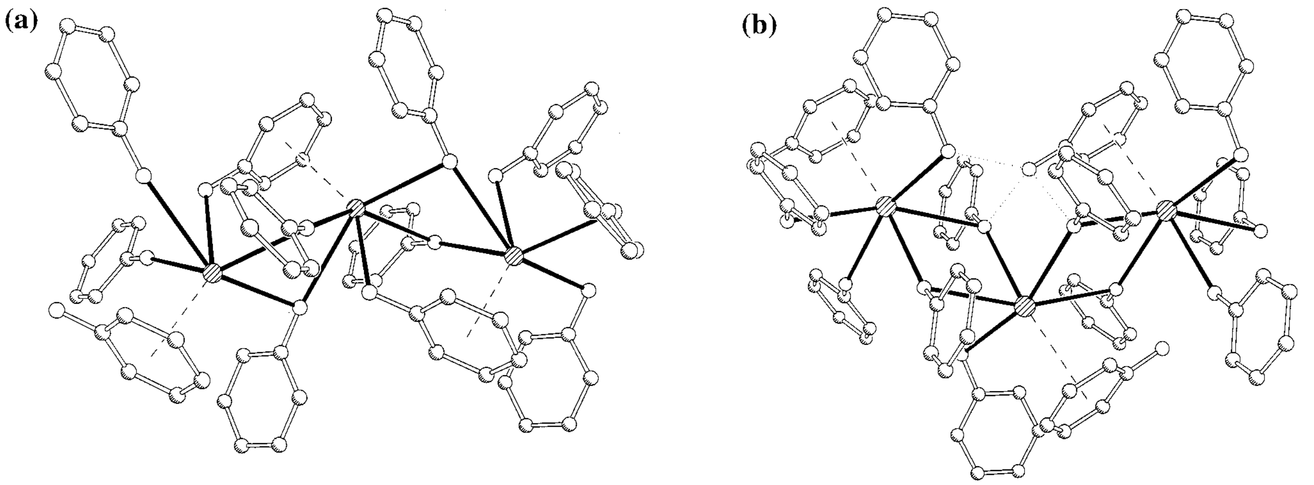

Figure 2. (a) Structure plot of orthorhombic $C_6H_5OK{\cdot}2(C_6H_5OH)$. Hydrogen atoms are omitted for clarity. The chain direction is along *c*. (b) Structure plot of orthorhombic $C_6H_5OK{\cdot}3(C_6H_5OH)$. Hydrogen atoms are omitted for clarity. The chain direction is along *c*.

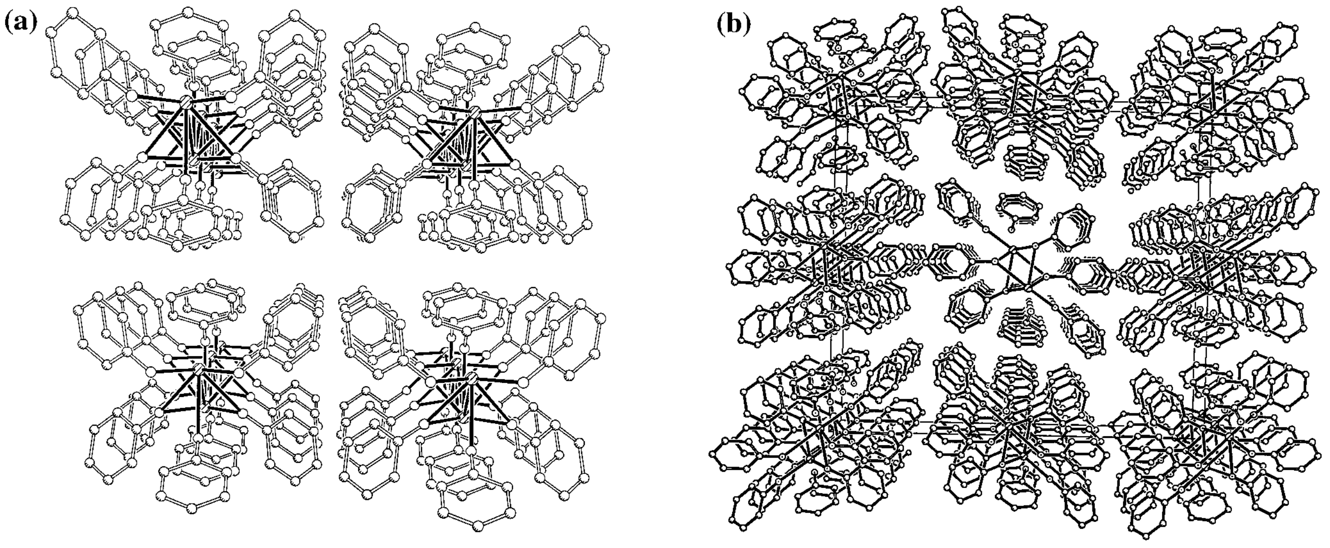

Figure 3. (a) Stacking plot of orthorhombic $C_6H_5OK{\cdot}2(C_6H_5OH)$. Hydrogen atoms are omitted for clarity. The chain direction is along *c*. (b) Stacking plot of orthorhombic $C_6H_5OK{\cdot}3(C_6H_5OH)$. Hydrogen atoms are omitted for clarity. The chain direction is along *c*.

and is comparable to that of **II** and C_6H_5OK. Interchain interaction is realized by nonbonded van der Waals forces only, whereas the phenyl rings of neighboring chains are interleaved (Figure 3a).

Compound **II** (Figure 2b) is more saturated with solvent and also has a structure of zigzag chains formed by polyhedra of potassium ions linked via edges, here in the cis position. Unlike in **I**, the potassium ion is coordinated in an almost perfect octahedron consisting of five oxygen atoms and the center of one phenyl ring (Figure 4). The general formula of the chain is $[K^{[6]}O_2^{[2]}O^{[1]}\pi(\text{phenyl})^{[1]}]$, which is of the same form as for **I**. However, in contrast to **I**, the phenolate oxygen atom is not linked to the potassium ion. It is connected to each phenol oxygen atom via hydrogen bonding (shortest O−O distance of 2.4 Å) which again strengthens the chain configuration. The packing density of 68.4% is about the same as for **I** (Figure 3b). As in the case of **I**, the positions of the three hydroxy hydrogen atoms of the phenols could not be found by powder methods. Despite this, the phenolate ion could be recognized by geometrical considerations. The more solvent molecules that are embodied in the structure, the stronger the tendency to solvate anion and cation separately. In structure **I** the phenolate oxygen atom takes part in the coordination besides the donation of the π electron system as ligand. In structure **II**, however, the cation coordinates the phenolate molecule solely via its π electron system. Furthermore, the formation of hydrogen bonds is evidently significant for the crystal structures of phenolates containing protic solvents. Short O(phenolate)−O(phenol) distances indicate hydrogen bonding in compounds **I** and **II**. A special feature of the alkali metal phenolate−phenol complexes is the coordination of the π electrons of the phenol molecules as well as of the phenolate ion by the metal ions.

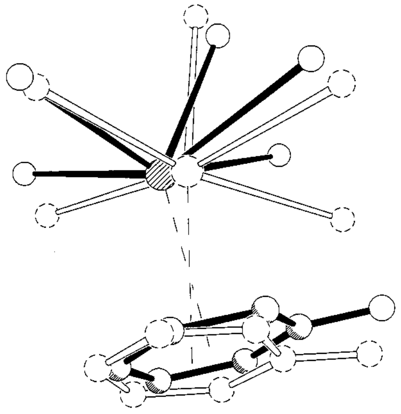

Figure 4. Comparison of coordination polyhedra of $C_6H_5OK{\cdot}2(C_6H_5OH)$ (solid bonds) and $C_6H_5OK{\cdot}3(C_6H_5OH)$. The positions of the potassium atom and of two oxygen atoms for each polyhedra have been matched by LSQ technique.

A comparison of the structures presented here and other alkali metal phenoxides leads to a classification of the coordinating solvent into two ligand groups. The coordination of aprotic ligands such as THF, NMC, dioxane, etc. results in structures of the heterocubane type[23,24] in contrast to protic ligands such as water, methanol, and as in our case phenol which lead to structures consisting of chains and layers.[22,25,26] The size of the ligands, the number of coordination partners, as well as the number of coordination positions of the ligand are important factors in determining the nature of the formed polyhedra and of the way they are linked. Small ligands such as water allow a two-dimensional network. Phenol, as a more bulky ligand usually permits only a one-dimensional linking to chains.

Entirely different structures have been found for other alkaline phenolate complexes containing phenol as solvent with Na and Cs as central cations. In $C_6H_5ONa{\cdot}2(C_6H_5OH)$,[21] two different distorted tetrahedral environments occur around the Na. One Na is coordinated by four oxygens deriving from phenols and the phenolate ion, and the other by three oxygens deriving from phenols and the phenolate ion and by the π electrons of the phenolate ion. The coordination polyhedra form chains of edge and corner sharing polyhedra. The larger cations K, Rb, and Cs have a 6-fold coordination. In $C_6H_5OCs{\cdot}2(C_6H_5OH)$,[27] the coordination sphere around the Cs cation consists of three oxygens and the π electrons of three phenyl rings, satisfying its steric requirements. The structure does not consist of edge sharing polyhedra but of pseudo-octahedra bridged by the μ^2-functionality of the ligands. In contrast, the structure of $C_6H_5ORb{\cdot}3(C_6H_5OH)$,[27] although having a different space group, is very similar to that of **II** and shows the same type of chain and coordination around the cation.

Conclusion

We have applied the technique of powder X-ray diffraction to solve the crystal structures of two moderately complex coordination compounds, containing 13 and 29 non-hydrogen atoms in the asymmetric unit. It is important for the community of solid-state chemists to recognize that these techniques are available, and are becoming routine. A general prerequisite to success in ab initio structure determination from powder data is a highly crystalline sample, coupled with high resolution data collection, to have as many independently measured reflections as possible. Even with this condition met, for moderately large unit cells, standard techniques such as direct methods often fail to solve the structure due to the overlap of unresolved peaks. The pseudo-atom technique used here is an extension to direct methods, applicable if there are several weakly scattering atoms in a chemically known configuration such as a phenol group.

There has been considerable recent progress in the use of powder diffraction to solve organic molecular crystals by real space search of a known structural fragment.[28] However, considering the computer power available, such techniques would not have been applicable to the present problem, because there was no useful prediction of the way in which the several phenols link together into the unit cell. The pseudo-atom method substitutes small rigid molecular fragments by spherically symmetric form factors. This simplification considerably reduces the degrees of freedom of rigid bodies at the beginning of a structure solution.

For an interpretation of the Kolbe–Schmitt synthesis, we may conclude the following: In chain structures as presented here, the phenolate molecule is situated in a position which can be attacked by carbon dioxide more easily than in layer structures observed for the phenolate–hydrate complexes. Furthermore, since it is proposed that the first step of carboxylation is an interaction between the alkali metal cation and the CO_2 molecule,[27,29] a contact between the cation and the phenolate anion should be a necessary condition for the carboxylation of the phenolate, which applies for compound **I**. Moreover, **I** and **II** show an interaction of the cation and the π electron system of the phenolate ion. This interaction supports the reactivity of the anion. Hence, small amounts of phenol, the presence of **I** or **II** besides C_6H_5OK, should not lead to a significant decrease of the amount of reaction product. This is indeed observed in carboxylation experiments carried out on the structurally related Na phenolates, C_6H_5ONa and $C_6H_5ONa{\cdot}2(C_6H_5OH)$.[29]

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Supporting Information Available: Tables listing atomic coordinates, figures of the coordination polyhedra of the potassium atoms, listings of selected bond distances and angles, and diffraction peak positions and intensities for $C_6H_5OK{\cdot}2(C_6H_5OH)$ and $C_6H_5OK{\cdot}3(C_6H_5OH)$ are available. (4 pages). Six ASCII files are also available on the Internet only. Ordering and access information is given on any current masthead page.

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