were interpreted as examples of increases in C–S bond lengths. However, the present result has brought down the C–S distances to values almost equal to the free molecule value. The Cu–S–C angles fall within 105° and 111°; that around S_{II}, which was 99°, has changed to a more reasonable value of 105°.

The ligands are all planar within experimental error and analyses of the anisotropic temperature factors reveal that the nitrogen atoms have the directions of the largest thermal vibration nearly perpendicular to the planes of the ligands. The central copper atom is about 0·7 Å away from the planes of ligands (II) and (III). (See Table 2.) Each NH_2 group is associated with one short NH–Cl contact; the N–Cl distances are between 3·24 and 3·44 Å and surround the chloride ion in a rather unusual way (*cf.* Fig. 5(*a*) and (*b*) of KOP(I)); the possible hydrogen-bond formation has been explained in the previous report. It should also be noted here that all N–N distances are longer than 3·4 Å and no N–H · · · N hydrogen bond is expected in the structure; in this structure, not all of the available hydrogens are involved in hydrogen-bond formation. As reported previously (KOP(I)), several N–S separations shorter than 3·4 Å are also found in the structure; according to a recent review paper by Wallwork (1962), these separations might be interpreted as N–H · · · S hydrogen bonds.

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Crystallographic data for $LiBr.2(CH_3)_3.COH.H_2O$, a lithium bromide-tertiary butyl alcohol complex.

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Spectral studies (Bufalini & Stern, 1961) of mixtures of alcohols and electrolytes suggest that, in many cases, strong interactions occur between the hydroxyl group of the alcohol and the ions in solution. Occasionally, the interactions are sufficiently strong to precipitate crystalline complexes.

A crystalline complex of lithium chloride or lithium bromide with tertiary butyl alcohol (TBA) is obtained by one of two methods: (1) by floating pure TBA on top of a saturated salt solution, whereupon crystals form at the interface; (2) by mixing the two liquids together, heating, and allowing them to cool to room temperature. By the second method, single crystals form in the less dense alcohol layer; large well-shaped crystals with distinct cleavage directions for LiBr and thin flexible platelets for LiCl. These crystals are stable in pure TBA, but decompose when placed in air, the saturated salt solution, water, benzene, dioxane, or hydrocarbons. It was possible, however, to determine the densities of the crystals by the flotation method in a mixture of TBA and carbon tetrachloride. These are: LiBr–TBA, 1·04 g.cm^{-3}; LiCl–TBA, 0·98 g.cm^{-3}.

The composition of the LiBr–TBA complex was obtained from a titration of the bromide ion and by heating the crystals and determining the percentage of alcohol in the distillate. The titration indicated that the crystals contain approximately 30% LiBr by weight, while the alcohol determination established the formula as $LiBr.2(CH_3)_3COH.H_2O$.

Only the LiBr complex was examined by X-rays, for the crystals are more stable than those of the LiCl complex which give a very poor diffraction pattern. Using a GE XRD 5 with Single Crystal Orienter and Cu $K\alpha$ radiation, we found that the LiBr complex crystallizes in the orthorhombic system with a *C*-centered cell. The lattice constants and conditions for reflections are:

a = 19·31 Å ∓ 0·01 Å *hkl* $h+k=2n$
b = 32·23 Å ∓ 0·01 Å *hk*0 $h=2n,\ k=2n$
c = 15·88 Å ∓ 0·01 Å

The space group, as determined from these conditions, is either *C2ma* or *Cmma*. From the formula, unit-cell size, and density, one finds that there are 24 formula weights in the unit cell (calculated density, 1·03 g.cm^{-3}). If the space group is *C2ma*, there can be three formula units in the asymmetric unit provided that none are in special positions, whereas *Cmma* requires at least some of these atoms to be in special positions. No further work is intended on either of these compounds.

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