

## Crystal Structure of Isobutyryl Hexachloroantimonate(V), $\text{Me}_2\text{CH}\cdot\text{CO}^+\text{SbCl}_6^-$

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THE EXISTENCE of oxocarbonium ions has been established by i.r. studies in the solid state, and n.m.r. methods in solution.<sup>1-3</sup>

The geometrical structure of the methyloxocarbonium ion,  $\text{MeCO}^+$ , has been determined by X-ray crystallography, in  $\text{SbF}_6^-\text{MeCO}^+$ <sup>4</sup> and  $\text{SbCl}_6^-\text{MeCO}^+$ .<sup>5</sup> No other structural determination of oxocarbonium ions has been reported so far.

We now report the crystal structure of  $\text{SbCl}_6^-\text{Me}_2\text{CH}\cdot\text{CO}^+$ . Some significant bond lengths found are shown in the Figure, their approximate

probably slightly weaker than the carbon-methyl bond in  $\text{Me}\cdot\text{CO}^+$ . The bond distance  $\text{Me}_2\text{CH}\cdot\text{CO}^+$  is 1.44 Å ( $\sigma = 0.02$  Å) whereas the methyl-carbon bond  $\text{Me}\cdot\text{CO}^+$  is 1.38 Å ( $\sigma = 0.02$  Å).<sup>4,5</sup> The difference of 0.06 Å is however not strictly significant.

The C-O distance of 1.11 Å is close to that of 1.12 Å found in  $\text{MeCO}^+$ .<sup>4,5</sup>

Crystals of  $\text{Me}_2\text{CH}\cdot\text{CO}^+\text{SbCl}_6^-$  have been prepared from chloroform solutions of the halides. Crystal data are:  $M = 421.38$ , orthorhombic.  $a = 19.850$  (20),  $b = 7.121$  (7),  $c = 9.194$  (9) Å,  $U = 1.299$  Å<sup>3</sup>,  $Z = 4$ ,  $D_0 = 2.11$ ; Space group  $Pnma$  (No. 62).

Intensities of 930 independent non-zero reflexions were recorded on a Pailred diffractometer using  $\text{Mo-K}\alpha$  radiation. The crystal ( $0.25 \times 0.25 \times 0.35$  mm.), very sensitive to moisture, was sealed in a Lindemann capillary tube. No absorption corrections were applied ( $\mu = 16.8$  cm.<sup>-1</sup>).

The structure was derived from the three dimensional Patterson function. The refinement to  $R = 0.066$  was carried out by isotropic full matrix least-squares analysis and difference synthesis.

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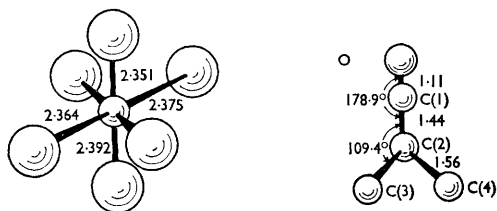


FIGURE.  $\text{SbCl}_6^-$  and  $\text{Me}_2\text{CH}\cdot\text{CO}^+$  ions in isobutyryl-hexachloroantimonate(V).

standard deviations being 0.02 for C(1)-C(2) and C(1)-O bonds, 0.03 for the C(2)-Me and 0.006 Å for the Sb-Cl bonds.

The carbon-isopropyl bond in  $\text{Me}_2\text{CH}\cdot\text{CO}^+$  is

<sup>1</sup> G. A. Olah, "Friedel-Crafts and Related Reactions" vol. 1, Interscience, New York, 1963, p. 91-95, 665-680, and 790-812.

<sup>2</sup> G. A. Olah, S. J. Kuhn, W. S. Tolgyesi, and E. B. Baker, *J. Amer. Chem. Soc.*, 1962, **84**, 2733.

<sup>3</sup> B. P. Susz and J. Wuhmann, *Helv. Chim. Acta*, 1957, **40**, 722, 971.

<sup>4</sup> F. P. Boer, *J. Amer. Chem. Soc.*, 1966, **88**, 1572.

<sup>5</sup> R. Weiss and J.-M. Le Carpentier, *Compt. rend.*, 1967, **265**, 797.