

THERMOCHEMICAL STUDY OF FULLERENE CHLORIDE $C_{60}Cl_{30}$

Lukyanova V.A., Papina T.S., Troyanov S.I., Buyanovskaya A.G.

Lomonosov Moscow State University, Chemistry Department,

Leninskie gory 1, bl.3, Moscow, 119992, Russia

⁽¹⁾A.N.Nesmeyanov Institute of Organoelement Compounds, Russian Academy of Sciences,

Vavilov str. 28, Moscow, 119991, Russia

Fax: 095-939-1240 E-mail: lukyanova@phys.chem.msu.ru

Halogenated fullerenes are of great interest as precursors for synthesis of various compounds having uncommon properties.

A novel, highly chlorinated derivative of fullerene, $C_{60}Cl_{30}$, was synthesized lately [1]. It was obtained by heating C_{60} with a large excess of $SbCl_5$ in glass ampoules at temperature 220-250^o C during 1-3 days. Then the ampoules were opened, and the excess of $SbCl_5$ and its decomposition products were removed by dissolution in HCl.

For the thermochemical study, about 450 mg of $C_{60}Cl_{30}$ were obtained in one synthetic run. The sample looked as dark-brown crystals. Results of X-ray analysis of the sample as well as good agreement of the experimentally obtained and theoretically calculated IR spectrum proved absence of impurities. Elemental analysis yielded C and Cl contents equal to 39.78 and 59.41 % correspondingly (the calculated ones are 40.39 and 59.61). These results confirm rather high purity of the sample.

This sample was used for measuring its combustion energy in a calorimeter with an isothermal jacket and a rotating bomb lined with platinum.

It is known that fullerenes and their derivatives burn badly and so calorimetric determination of their combustion enthalpy is a complicated experimental task.

In the present work conditions ensuring complete combustion of fullerene chloride $C_{60}Cl_{30}$ were worked out. For this purpose small quantities of the sample (about 40 mg) were burned together with an auxiliary substance (benzoic acid) at high pressure of oxygen in the bomb (4.05 MPa). A weighed quantity of the sample was sealed into a

terylene-film bag; it was placed on platinum-wire netting, fixed over a thin-walled platinum crucible, between two pellets of benzoic acid (about 360 and 100 mg). Such location ensured the best access of oxygen to the substances. Under these conditions complete combustion of the fullerene chloride was achieved.

10 ml of 0.1 N As_2O_3 solution was placed into the bomb before the experiment to reduce free chlorine (formed during combustion of fullerene chloride) to HCl. The temperature rise was measured with a copper resistance thermometer and a bridge circuit. The products of combustions were analyzed for contents of $CO_2(g)$, $As_2O_3(solution)$ and $H_2PtCl_6(solution)$. The absence of CO was checked using indicator tubes.

The standard values of combustion (-24456 ± 135 kJ/mole) and formation (133 ± 135 kJ/mole) enthalpies of $C_{60}Cl_{30}$ were determined from the results of calorimetric experiments.

The work was supported by RFBR, grant 03-03-33138.

References

- 1.Troshin P.A., Lyubovskaya R.N., Ioffe I.N., Shustova N.B., Kemnitz E., Troyanov S.I. Synthesis and Structure of the Highly Chlorinated [60]fullerene $C_{60}Cl_{30}$ with a Drum-Shaped Carbon Cage. *Angev.Chem.Int.Ed.* 2005; 44:234-237.
- 2.Sidorov L.N., Yurovskaya M.A., Borshchevskij A.Ya., Trushkov I.V., Ioffe I.N. Fullerenes. : Moscow: Examen, 2004. p.184-186, 251-252.