Isolation and characterisation of $C_{60}F_{38}$

Joan M. Street,[a] Ala'a K. Abdul-Sada,[b] Olga Boltalina[c]
Brian W. Clare,[d] David L. Kepert,[d] and Roger Taylor[b]

[a] Chemistry Department, The University, Southampton, SO17 1BJ, UK

[b] The Chemistry Laboratory, CPES School, Sussex University, Brighton BN1 9QJ, UK

[c] Chemistry Department, Moscow State University, Moscow 119899, Russia.

[d] Biomedical and Chemical Sciences School, University of Western Australia, Crawley, Australia WA 6009

Mixtures of highly fluorinated fullerenes $C_{60}F_n$ ($36<n<48$) are readily obtained in many fluorinations, however their separation into specific compounds is difficult due to similarity of their properties. Structural data have been reported for only two fluorofullerenes in this series *viz.* $C_{60}F_{48}$[1] and $C_{60}F_{36}$[2]. Fluorination with AgF_2 affords $C_{60}F_{44}$,[3] but its structure is yet to be determined.

Isolation of the specific fluorofullerenes with high F content and narrow distribution of the products can be achieved by fluorination with metal fluorides either alone or in combination. Thus, from fluorination of [60]fullerene either with MnF_3 at 330 ^{o}C or with MnF_3/K_2NiF_6 at 480 ^{o}C we have isolated two isomers of $C_{60}F_{38}$, each of which elute (HPLC) faster than any isomer of $C_{60}F_{36}$ The quantity of one isomer was too small for structural identification, but analysis of both the 1 D and 2 D ^{19}F NMR spectra of the other (which has C_1 symmetry), coupled with density functional calculations, has identified possible structures from amongst the many *millions* of possibilities

We shall describe how the content of $C_{60}F_{38}$ in the MnF_3-samples that have $C_{60}F_{36}$ as a major product, can be significantly enhanced by varying reaction conditions. It is hoped in this way to obtain large samples suitable for single crystal x-ray analysis.

[1] A. A. Gakh, A. A. Tuinman, J. L. Adcock, R. A. Sachleben and R. A. Compton, J. Am. Chem. Soc., 1994, **116**, 629; V. I. Privalov, O. V. Boltalina, N. A. Galeva and R. Taylor, *Doklady Chem.*, 1998, **360**, 499; S. I. Troyanov, P. A. Troshin, O. V. Boltalina, I. N. Ioffe and L. N. Sidorov, E. Kemnitz, Angew. Chem., Int. Ed. 2001, **40**, 2285.

[2] O. V. Boltalina, J. M. Street and R. Taylor, J. Chem. Soc., Perkin Trans. 2 1998, 649; P. B. Hitchcock and R. Taylor, *Chem. Commun*., 2002, 2078; A. G. Avent, B. W. Clare, P. B. Hitchcock, D. L. Kepert and R. Taylor, *Chem. Commun*., 2002, 2370.

[3] A. A. Goryunkov, Yu. V. Markov, O. V. Boltalina, B. Zemva and R. Taylor, J. Fluorine Chem. 2001, **112**, 191-196.