Degradation of C_{60} by light

SIR – Using production, extraction and purification procedures published previously[1,2], we have prepared numerous batches of pure C_{60}. Freshly prepared samples dissolve readily in benzene to give the deep magenta solutions that we have described[2], but samples stored without exclusion of light or oxygen will not redissolve completely – a reddish-brown deposit remains. Moreover, benzene solutions of C_{60} exposed to light slowly produce a small amount of brown deposit. Digestion with benzene of solid samples of pure C_{60} that have been stored in this way leads to successively paler extracts with an increasingly pinkish hue, we believe owing to the presence of traces of the decomposition product.

We have also observed that during column chromatography of mixtures of C_{60} and C_{70} on neutral alumina using hexane as eluent, the magenta colour from C_{60} on the column is gradually lost if elution is interrupted and the column exposed to light. Both observations suggest the occurrence of some kind of photochemical reaction. We have confirmed this by irradiation of solutions of C_{60} in HPLC-grade hexane with a water-cooled medium-pressure silica-jacketed Hanovia insertion ultraviolet lamp. We monitored the disappearance of C_{60} by ultraviolet spectroscopy; reaction is complete within 10–16 hours. A buff precipitate is formed which consists of a mixture of products possessing the following features.

(1) Mass spectroscopy indicates that the fullerene cage is substantially or completely fragmented, depending on the reaction conditions. Fragmentation is less if the reaction is carried out under nitrogen (whence the oxygen content of the solution is reduced but remains significant). The products are not polymeric.

(2) The unprocessed product typically has a hydrogen content of 3–4 per cent and an oxygen content (determined by difference) of 40–54 per cent, depending on conditions; nitrogen is absent. It dissolves readily in polar solvents.

(3) Infrared and NMR spectra indicate that the following groups are present: C–H (including aromatic C–H), C=O (very intense), C–O, C=C, substituted aromatic or alkene, C≡C, C_2H_5, CH_2, alkyl–O, and CH_2–O (substantial). The product is partly soluble in potassium bicarbonate, indicating the presence of a carboxyl group.

Confirmation that hydrogen is abstracted from the solvent was provided by irradiation of a solution of C_{60} in benzene, reaction being slower. Infrared spectra indicated the absence of OH groups, but C–H (including aromatic) and C=O bands were found.

Irradiation of a C_{60} solution in hexane with a glass-enveloped medium-pressure lamp resulted in only trivial loss of colour after 16 hours, confirming that photochemical degradation requires ultraviolet irradiation.

Five conclusions follow from our work. First, aldehydes and/or ketones are formed, implying reaction with ozone. Given the accepted structure of the intermediate ozonide[3], oxygen may end up within the cage, before cleavage to give the product.

Second, C_{60} may be ultraviolet-sensitive in the absence of oxygen, and may thus partly decompose in the reaction vessel in which it is prepared (see, for example, refs 4,5) owing to the intense ultraviolet irradiation emanating from the carbon arc. Substantially higher yields of C_{60} may therefore be achievable by using a flow system.

Third, samples of C_{60} should be stored in the dark and under vacuum or nitrogen. Solutions of C_{60}, especially in either alkanes or cycloalkanes, should also be kept in the dark.

Fourth, although the alkyl–oxygen bonds present may in part arise from epoxides, NMR results show that three-membered carbon chains at least are also involved, and the most probable source of these is the solvent. Use of photochemical irradiation and suitable co-reagents may therefore lead to a whole range of derivatives of C_{60}.

Finally, the reason for the failure hitherto to observe naturally occurring C_{60} on Earth now becomes clear. (We have also failed to find it in chimney soot.)

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Discrepancies in AIDS virus data

SIR – In early 1983 we described a new human retrovirus, then called LAV, now called HIV, associated with lymphadenopathy in a patient at risk of AIDS[1]. We tried to propagate this isolate in continuous cell lines such as CEM and B-lymphoblastoid cell-like RAJI without any success, as reported at the first European conference on AIDS in Naples on 25 June 1983 and subsequently published[2]. Clearly, this virus would grow only in peripheral blood lymphocytes (PBL), in cord blood lymphocytes and in bone marrow monucleated cells.

We propagated this isolate exclusively in PBL for more than a year, and in 1984, we adapted this virus to Epstein–Barr virus-transformed B-cell line[3]. The virus was subsequently cloned[4] and sequenced[5]. Nothing was known about the genomic variability of various HIV isolates in 1983.

We found several discrepancies concerning the origin and the growth of this virus either in PBL or in continuous cell lines. Molecular analysis of sequence from previous isolates showed differences between the cloned and sequenced HIV[6]. We suggested that at least two variants could be present in the PBL-passed BRU and decided to analyse the samples biologically as well as molecularly. We used the HIV-BRU virus (JBB–LAV, January 1984) continuously propagated in PBL from an HIV-1-negative donor and took the sample maintained from June 1983 to January 1984 in these cells.

BIOLOGICAL PROPERTIES OF ISOLATES DERIVED AFTER GROWTH IN INDICATED CELLS

	BRU PBL 84 (JBB LAV)	Early passage of JBB–LAV in CEM	LAV 90 continuous passage in CEM	Molecular clone of LAV (transfected)
OKT4 A in PBL (5 μg ml^{-1})	No	No growth	Yes	Yes
OKT4 A in CEM (5 μg ml^{-1})	Yes	Yes	Yes	Yes
Soluble CD4 in PBL (5 μg ml^{-1})	No	+/–	Yes	Yes
Size of syncytia in MT4	Small	Normal	Large	Small
Neutralizing antibodies to HIV1–LAV	No	Yes	Yes	Yes
V3 loop (RIPA)	Absent	Present	Present	Present
$TCID_{50}$ in PBL	10^{-5}	$< 10^{-1}$ No growth	10^{-1}	
CEM	$<10^{-1}$	10^{-2}	10^{-3}	
MT4	10^{-5}	10^{-1}	10^{-3}	

Virus stocks, titration pattern, block of the entry with OKT4 A or soluble CD4 are described in ref. 10; neutralizing-antibody determination in ref. 11. OKT4 A (Ortho Diagnostics), soluble CD4 was obtained from ANRS (D. Klatzmann). Monoclonal antibodies against V3 were from ANRS hybridolab Institut Pasteur (J. C. Mazie).
No, no block of virus growth by OKT4 A or soluble CD4 neither neutralization by antibodies.
Yes, block and neutralization.