

Polyhydrogenation of [60]- and [70]-fullerenes

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Reduction with Zn–conc. HCl in either benzene or toluene solution, results in rapid and quantitative conversion of [60]- and [70]-fullerenes into mainly C₆₀H₃₆ and C₇₀H_{36/38}. Significant amounts of more highly hydrogenated derivatives are also formed. Mass spectra under EI conditions can be obtained free of peaks due to either less-hydrogenated species or the parent fullerenes, provided they are obtained immediately, since both compounds undergo rapid light-catalysed degradation in the presence of oxygen, to give the parent fullerenes, oxygen-containing derivatives (fullerenols) and lower hydrides; C₆₀H₁₈ is the main product from C₆₀H₃₆. Formation of reduced fullerenes up to C₆₀D₄₄ and C₇₀D₄₈ on reaction of [60]- and [70]-fullerenes with Zn–conc. DCl, is attributed to the higher stability of C–D compared with C–H bonds, which provides greater compensation for the loss of resonance energy and the greater steric compression that accompanies reduction beyond the 36 H level. Laser-desorption time-of-flight mass spectrometry indicates that the absence of the corresponding higher hydrides (as opposed to deuterides) is not due to decomposition during EI mass spectrometry. The hydrides do not undergo hydrogen exchange with D₂O either alone or in the presence of either sodium hydrogen carbonate or sodium hydroxide. C₆₀H₃₆ has considerable thermal stability but that for C₇₀H_{36/38} is lower. HPLC chromatograms, as well as IR, UV–VIS, ¹H NMR, and mass spectra have been obtained for both compounds. Each appears to be highly resistant to further reduction by hydrogen–catalyst, but shows a surprising tendency to form trimethylene adducts, by an unknown mechanism.

Speculations concerning the possible use of fullerene hydrides in batteries have appeared in numerous media articles. Both the properties of fullerene hydrides and methods suitable for their large-scale preparation are of considerable interest. C₆₀H₃₆, the first fullerene derivative to be prepared, was obtained under Birch reduction conditions;¹ C₆₀H₁₈ was a secondary product. The ¹H NMR spectrum of the product mixture was reported to show a broad band between δ 4.2 and 2.5 with maxima at δ 3.80 and 3.35. The structure proposed for C₆₀H₃₆ had all of the remaining twelve double bonds isolated (in pentagons which is unfavourable).² Subsequently a tetrahedral (*T*) structure was proposed containing four highly delocalised benzenoid rings, a reason for reduction to stop at this stage;³ subsequent calculations support this proposal.⁴

A later mass spectroscopic study of Birch reduction of [60]fullerene indicated the formation of polyhydro[60]fullerenes ranging from C₆₀H₁₈ to C₆₀H₃₆ with the distribution centred on C₆₀H₃₂.⁵ The variance between these and the earlier results was attributed to the different mass spectroscopic techniques employed, and to the thermal instability of the fullerene hydrides. Both C₆₀H₁₈ and C₆₀H₃₆ have been produced from [60]fullerene by hydrogen transfer from 9,10-dihydroanthracene.⁶ Transfer hydrogenation to [60]- and [70]-fullerenes from [7]benzanthrene, gave a very broad spectrum of polyhydrofullerenes centred on C₆₀H₃₆ and C₇₀H₃₆.⁷ C₆₀H₃₆ and C₇₀H₃₆ were also obtained by hydrogen radical-induced hydrogenation.⁸ Reduction of [60]fullerene by hydrogen with rhodium–alumina catalyst at ambient temperature and pressure gave mainly C₆₀H₂.⁹ With hydrogen and ruthenium–carbon as catalyst, temperatures up to 280 °C, pressures up to 130 atm and reaction times up to 24 h, a range of hydrogenated products *viz.*, C₆₀H_{2–12}, C₆₀H₁₈, C₆₀H_{36–50}, C₇₀H₄ and C₇₀H_{36–38} were obtained; on using Pd–C as catalyst some C₆₀H₃₆O was also formed.¹⁰ The ¹H NMR spectrum of C₆₀H₃₆ thus produced closely resembled that reported above, but surprisingly, so did the ¹H NMR spectrum for

C₇₀H_{36–38}.¹⁰ These workers also reported that whereas C₆₀H₁₈ and C₆₀H₃₆ decomposed slowly in nitrogen, degradation of C₆₀H₃₆ occurred suddenly in air.¹¹

Whilst the accumulated studies confirm that the structure of C₆₀H₃₆ is not dependent upon the Birch reduction conditions (indeed the Birch mechanism cannot give this structure), it has proved difficult to obtain satisfactory mass or ¹H NMR spectra for the product. We now address these problems, identify some underlying causes, and describe a simple and very rapid method easily adapted to the production (in principle) of kilogram quantities of C₆₀H₃₆, and C₇₀H_{36/38} mixtures per day. Previously we showed that all isomers of C₆₀H₂, C₆₀H₄, C₇₀H₂ and C₇₀H₄ resulting from 1,2-addition across high-order bonds could be obtained by diimide reduction of the corresponding fullerenes.¹² We now report the results of reduction of [60]- and [70]-fullerene by Zn–conc. HCl. Our results for [60]fullerene reduction differ substantially from those described (whilst our work was in progress) by Meier *et al.* who used Zn–6 mol dm⁻³ HCl and obtained a mixture of 7% C₆₀H₂, 3% C₆₀H₄ and 1% C₆₀H₆.¹³

Experimental, results and discussion

Reduction

(a) [60]Fullerene. Zinc (excess, granules) was added to a saturated solution of [60]fullerene in either benzene or toluene maintained under a nitrogen atmosphere with exclusion of light, at room temperature. Conc. hydrochloric acid was then added over 5 min to the rapidly stirred mixture and stirring was continued for a further 15 min. The colour of [60]fullerene was quickly lost to give a light-brown solution, which upon being stirred for a further 1 h, became colourless.

The reduction progress was followed by HPLC using a 25 cm × 4.5 mm Cosmosil column, operated at 23 bar. Samples were injected as toluene solutions and elution was with toluene (0.25 cm³ min⁻¹). The HPLC trace (285 nm) for the light-brown

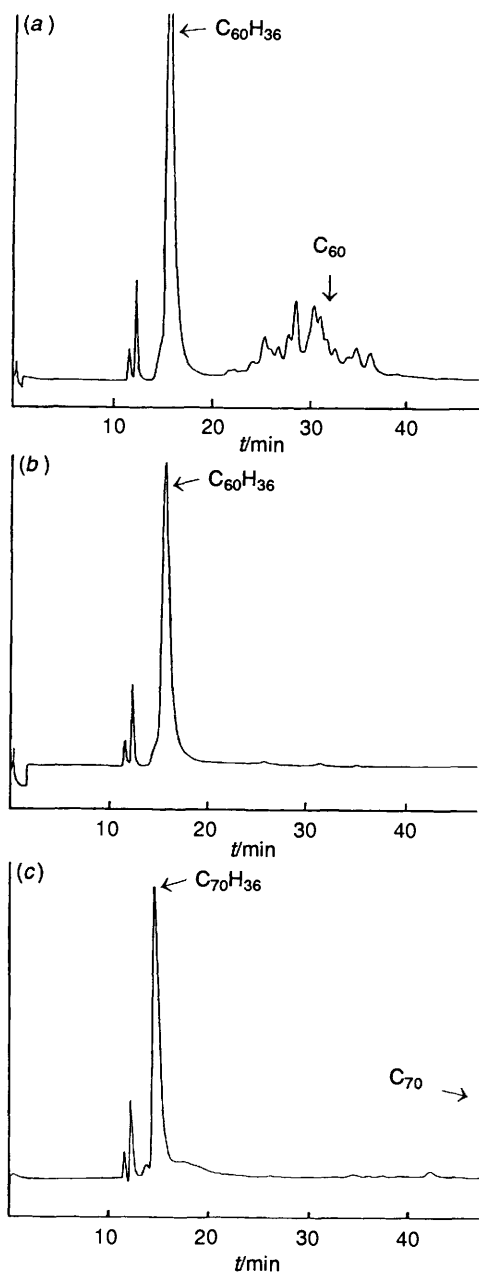


Fig. 1 HPLC traces (for conditions see the text) for: (a) the product of reduction of [60]fullerene in benzene by Zn-HCl, taken after 5 min reaction time; (b) the product of reduction of [60]fullerene in benzene by Zn-HCl, taken after 1 h reaction time; (c) the product of reduction of [70]fullerene in benzene by Zn-HCl, taken after 1.5 h reaction time

product [Fig. 1(a)] consisted mainly of a single peak (shown by mass spectrometry to be mainly $C_{60}H_{36}$) together with other products believed to be less-highly hydrogenated [60]fullerenes (the early peaks are due to the solvent front and to the pressure change upon injection). On this column, the retention time of $C_{60}H_{36}$ (15.5 min) is less than that of [60]fullerene (31.1 min); early elution of 1,2-dihydro[60]fullerene from a Phenomenex 'buckysep' column has also been reported.⁹ The HPLC trace for the colourless material showed that $C_{60}H_{36}$ was essentially free of less hydrogenated material [Fig. 1(b)].

Because fullerenes tenaciously retain solvent, and in order to minimise manipulations of the product, reduction for UV analysis was carried out using a saturated solution in hexane: $\lambda_{\max}(\text{hexane})/\text{nm}$ 207, 257, 274.5, 276.5, 283.5, 292.5, 300.5, 304.5, 307, 313, 317.5, 320.5 and 329 [Fig. 2(a)]; $\nu(\text{KBr})/\text{cm}^{-1}$

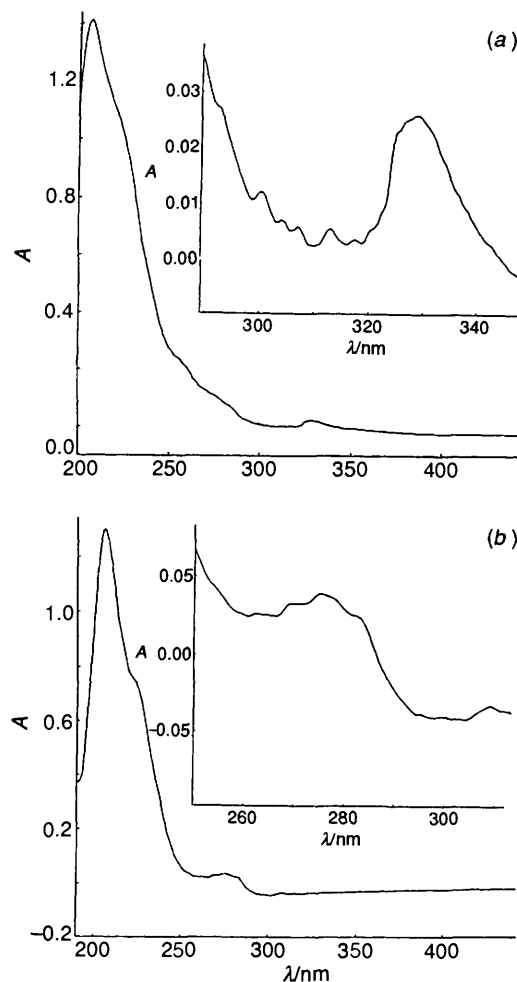


Fig. 2 UV spectra for (a) $C_{60}H_{36}$; (b) $C_{70}H_{36/38}$. Some higher hydrogenated species are present in each material as minor components.

2913, 2849, 1725, 1635, 1456, 1384, 1273, 1123, 1073, 1035, 730 and 678 [Fig. 3(a)].

Reduction with Zn-conc. DCl, used a [2H_6]benzene solution of [60]fullerene as above, and was complete after 1.5 h.

(b) [70]Fullerene. Previously we found that reduction of [70]fullerene with diimide (to give tetrahydro derivatives) took place less readily than the corresponding reduction of [60]fullerene.¹² This reactivity difference extended to the Zn-HCl reduction, which was complete after *ca.* 1.5 h, and gave a pale yellow product. The HPLC trace of this product (conditions as above) is shown in Fig. 1(c), and it is noteworthy that the retention time (14.7 min) is *less* than that for $C_{60}H_{36}$; by contrast, the retention time of [70]fullerene (*ca.* 62 min) is much greater than that for [60]fullerene on the column described. Reduction in order to obtain the UV spectrum was carried out in hexane, as above. $\lambda_{\max}(\text{hexane})/\text{nm}$ 207, 223.5, 255, 259, 263, 269.5, 275.5, 278.5, 283, 295.5, 300, 303, 309.5 and 312.5 [Fig. 2(b)]; $\nu(\text{KBr})/\text{cm}^{-1}$ 2909, 2852, 1730, 1456, 1282, 1126, 1035, 750, 730, 695, 587 and 468 [Fig. 3(b)].

Reduction with Zn-conc. DCl was carried out using a [2H_8]toluene solution of [70]fullerene as above.

In order to avoid proton interference, reductions to give samples suitable for NMR spectra were carried out in deuteriated benzene or toluene. The alternative of using ordinary solvents, removal of them after hydrogenation, and then attempting to redissolve the product in a small amount of deuteriated solvent was unsatisfactory, owing to the rapid formation of fullerlenols during the evaporation procedure.

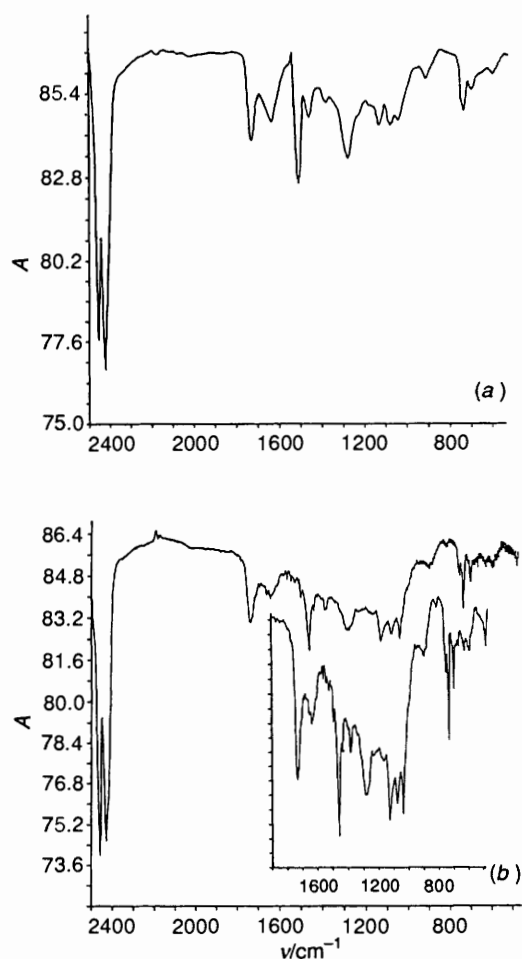


Fig. 3 IR spectra for (a) $C_{60}H_{36}$; (b) $C_{70}H_{36/38}$. Some higher hydrogenated species are present in each material as minor components.

Mass spectra

(a) Hydrogen-reduced [60]fullerene. Initial attempts to obtain mass spectra (of samples that were not reduced under nitrogen) gave results no better than those reported previously using other reduction techniques (see the introduction), *i.e.*, $C_{60}H_{36}$, $C_{60}H_{18}$, C_{60} , as well as oxygen-containing components were present. Earlier, we had found that reduction of [60]fullerene with H_2 -Pt-C gave a product which, after removal of the benzene solvent, would then only redissolve in either water, HCl or pyridine. We attributed this fact to allylic autoxidation of C-H bonds (giving fullereneols);¹² a similar reaction accounted for fullereneol formation during hydroboration of [60]fullerene.¹⁴ Both [60]- and [70]-fullerenes catalyse allylic oxidation of alkenes through the intermediacy of singlet oxygen, a process inhibited by the addition of DABCO (1,4-diazabicyclo[2,2,2]-octane).¹⁵ Likewise we found that the degradation of $C_{60}H_{36}$ (which is manifested by precipitation from benzene or toluene solution) could be retarded by the addition of Galvinoxyl.

Reasoning that the failure to obtain satisfactory mass spectra might be due to oxidation, we carried out reduction under a nitrogen atmosphere in the absence of light, the mass spectra (VG Autospec, EI conditions, 70 eV, 8 kV accelerating voltage) being run *immediately* after work-up. Excellent spectra were then obtained, showing the presence of $C_{60}H_{36}$ (with only a trace of the oxide, a recurring feature of fullerene mass spectra), and no [60]fullerene; Fig. 4(a) is typical.

By comparing the peak intensities of this spectrum with that simulated for $C_{60}H_{36}$ (inset) one may calculate (approximately because of the interference from the fragmentation ions) that

there is also present 15% of $C_{60}H_{38}$ and 3% of $C_{60}H_{40}$ ($C_{60}H_{36} = 82\%$); in another sample these values were 21% and 6%, respectively ($C_{60}H_{36} = 73\%$). By contrast, after HPLC purification of the sample (which removed $C_{60}H_{38}$ and $C_{60}H_{40}$) the peak intensities at 756, 757, 758, 759 and 760 amu became 100, 51, 18, 5, 1, in good agreement with those required for $C_{60}H_{36}$, *viz.*, 100, 67, 22, 5, 0. Because of the sensitivity of $C_{60}H_{36}$ to light and oxygen, some degradation occurs on processing so that lower hydrides and C_{60} appear in the spectrum.

(b) Deuterium-reduced [60]fullerene. Reduction using zinc-conc. DCl gave the mass spectrum shown in Fig. 5(a) (odd-numbered masses are now obtained because of rounding up, *e.g.*, m/z for $C_{60}D_{42}$ is calculated to be 804.59). Here not only are more highly hydrogenated species evident ($C_{60}D_{36}$, $C_{60}D_{38}$, $C_{60}D_{40}$, $C_{60}D_{42}$ and $C_{60}D_{44}$ are present in approximate respective concentrations of 32, 31, 22, 11, 4%), but the concentration of, *e.g.*, $C_{60}D_{38}$ relative to $C_{60}D_{36}$ is considerably higher than that for $C_{60}H_{38}$ compared with $C_{60}H_{36}$ described above. Whilst the difference could in principle be attributed to the greater stability of the deuterated species towards EI mass spectrometry, this interpretation can be discounted on two grounds. First, the concentrations of degradation products obtained from the hydrogenated species are no greater than those for the deuterated material [compare Figs. 4(a) and 5(a)]. Secondly, matrix-assisted laser desorption time-of-flight (MALDI-TOF) mass spectrometry (GSG focus linear instrument, 337 nm laser, 20 kV accelerating voltage, external calibration, sinapic acidmat) showed concentrations of $C_{60}H_{38}$ and $C_{60}H_{40}$ no higher than did the EI mass spectrum. The most probable explanation is that reduction of $C_{60}H_{36}$ requires the resonance energy of the benzenoid rings postulated in the *T* structure^{3,4} to be overcome, as well as increasing eclipsing strain in the cage; the higher C-D *vs.* C-H bond energy makes this more favourable in the deuterated compound.

The effect of the greater strength of the C-D bond compared with the C-H bond parallels fluorination, though in a less marked way. Although the fluorinated products suffer from much greater eclipsing strain, the high C-F bond energy makes it possible to add up to 60 fluorines to the cage, the species of highest concentration commonly obtained being $C_{60}F_{48}$.¹⁶

(c) Hydrogen-reduced [70]fullerene. Using the above procedure, good mass spectra [Figs. 4(b)-(d), three different product batches] were obtained for reduced [70]fullerene. Either $C_{70}H_{36}$ or $C_{70}H_{38}$ is the major component depending on the run, accompanied in some cases by $C_{70}H_{40}$, $C_{70}H_{42}$ (and possibly $C_{70}H_{44}$). For example, after allowing for the concentrations of the isotope peaks, the mass spectrum shown in Fig. 4(c) indicates approximate respective concentrations for $C_{70}H_{36-44}$ of 44, 28, 19, 7, 2%. Appreciable quantities of both $C_{70}H_{38}$ and $C_{70}H_{40}$ are produced, as found also using high pressure catalytic reduction.^{7,10} The yield discontinuity between $C_{70}H_{40}$ and $C_{70}H_{42}$ is also evident on similar treatment of the data shown in Fig. 4(d) and is consistent with the postulated aromatic nature of the product.¹⁷ The spectra reveal a greater degree of dehydrogenation than for $C_{60}H_{36}$, attributed to lower thermal stability (see below).

Fig. 4(b) also shows the presence of $C_{70}H_{36}O$ at $m/z = 892$; the peaks centred at $m/z = 918$ and 960 are discussed under trimethylene adducts, below.

(d) Deuterium-reduced [70]fullerene. Reduction of [70]fullerene by Zn-DCl gave the mass spectrum shown in Fig. 5(b). Here $C_{70}D_{36}$ is a minor component, $C_{70}D_{42}$ is dominant, and species up to $C_{70}D_{48}$ can be detected, the percentage yields of $C_{70}D_{36-48}$ being approximately 9, 13, 19, 22, 21, 13, 3%. The pattern therefore parallels that observed in reduction of [60]fullerene.

The effect of heat. $C_{60}H_{36}$ has good stability towards high

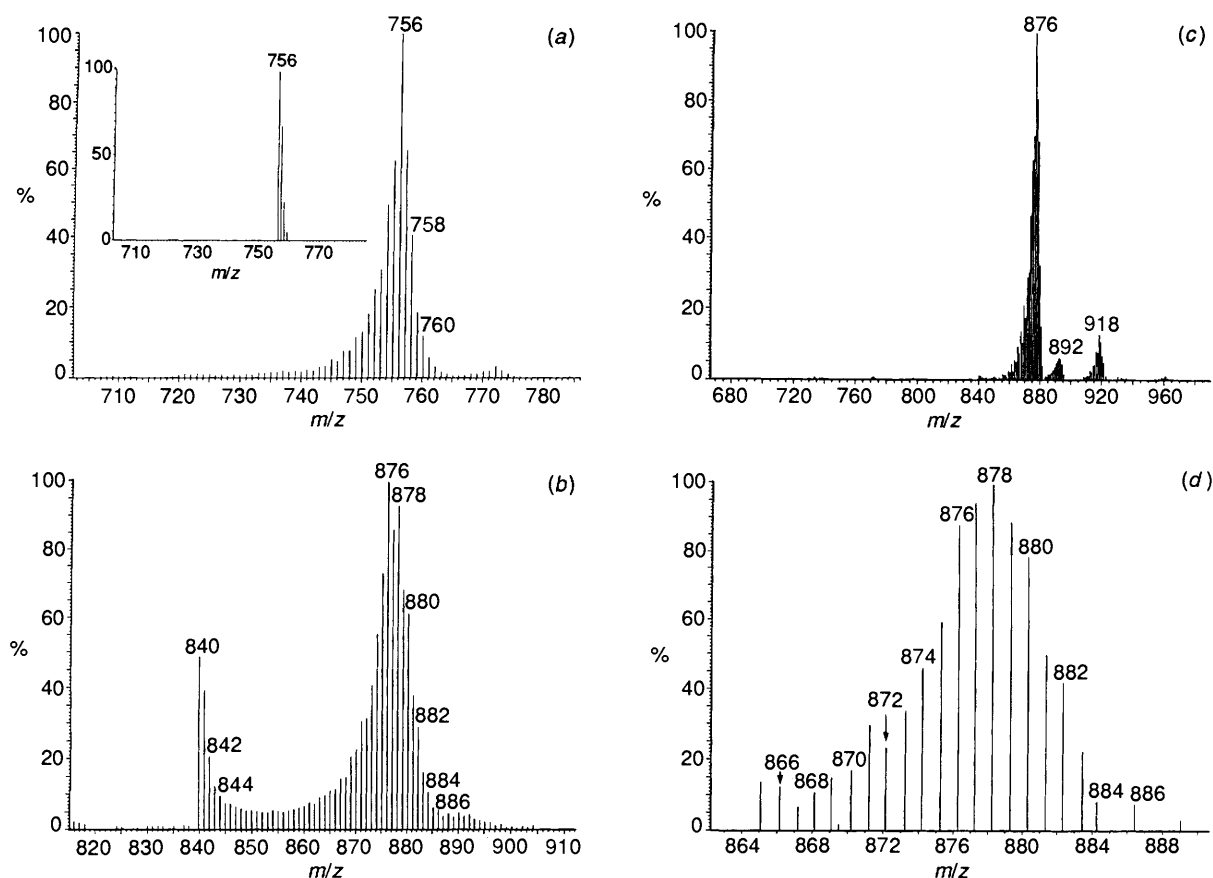


Fig. 4 Mass spectrum (EI conditions) for: (a) the product of reduction of [60]fullerene by Zn-conc. HCl (inset shows simulated spectrum for $C_{60}H_{36}$); (b)–(d) three products (different scale expansions) from reduction of [70]fullerene by Zn-conc. HCl

temperature. Figs. 6(a), (b) show, respectively, spectra taken initially and after being on a DCI probe at 650 °C for 9 mins. Slight enhancement of the $C_{60}H_{18}$ peak following heat treatment can be discerned, and this gradually becomes more severe after extended periods [Fig. 6(c)]; eventually complete degradation to [60]fullerene occurs.

$C_{70}H_{36}$ has lower thermal stability, and Fig. 7 shows the mass spectrum of the sample after 1 min on a DCI probe at 650 °C.

1H NMR spectra

No 1H NMR spectra have been published for either $C_{60}H_{36}$ or $C_{70}H_{36}$. Reports of broad bands for each are anomalous since (for the former at least) a simple spectrum (two doublets, one triplet) should be obtained. However, our present results suggest that the anomaly has a twofold origin. First, it is clear that the reported $C_{60}H_{36}$ compounds are, as in this work, a mixture of components hydrogenated to a greater (and possibly lesser) extent than $C_{60}H_{36}$. We attempted to overcome this difficulty by HPLC processing the reduced product, but although this removed the more highly hydrogenated material, decomposition products were introduced during the manipulations. Secondly, we have found that, owing to rapid oxidative degradation, it proved impossible to obtain a 1H NMR spectrum unless samples were sealed under either nitrogen or hydrogen, and the spectra run immediately. Even so, the long time required for data acquisition from the (necessarily dilute) deuterated toluene solutions resulted in some decomposition with precipitation (possibly from dissolved oxygen). Attempts to overcome this problem by using more concentrated solutions (in CS_2) were frustrated because the hydrogenated fullerenes reacted in this solvent. This also prevented our obtaining ^{13}C NMR spectra because of the long acquisition times needed.

(a) Reduced [60]fullerene. The 1H NMR spectrum [Fig. 8(a)] showed a broad band between δ 2.5 and 4.2 with peaks evident at δ 2.55, 2.95, 3.05, 3.3, 3.5, 3.9 and 4.0. These features, with varying degrees of sharpness, were duplicated in a number of samples, especially the 'double-hump', the hump centred at *ca.* δ 3.5 being always larger than that centred at *ca.* δ 3.0.

Apart from the reasons noted above, two other possible causes for the anomalous spectrum need to be addressed. One is long-range coupling which we attempted to overcome by reducing [60]fullerene with a mixture of DCI containing only a few percent of HCl. However, this strategy was unsuccessful because the mass spectrum of the product (m/z 760–800, maximum intensity at 781 amu) revealed that a very broad spectrum of products was obtained. The second possible cause is the lability of the hydrogens, and it is significant that treatment of one sample (which had been allowed to stand for a few days) with Galvinoxyl radical inhibitor caused both the NMR and mass spectrum to disappear; the latter showed the presence of [60]fullerene only. However, Galvinoxyl had no effect on freshly-prepared material, neither did iodine or *p*-benzoquinone.

(b) Reduced [70]fullerene. The difficulties noted above also applied to hydrogenated [70]fullerene. A broad band between δ 2.4 and 4.0 was observed Fig. 8(b), though with a different profile from that obtained with $C_{60}H_{36}$. The more upfield location of the spectrum compared with that for $C_{60}H_{36}$ is consistent with the pattern noted for the di- and tetra-hydro derivatives.¹²

The effect of light and oxygen

The sensitivity of the hydrogenated fullerenes to air is greatly enhanced by light. Samples maintained in air, but in the dark are reasonably stable. Samples of $C_{60}H_{36}$ kept in the dark

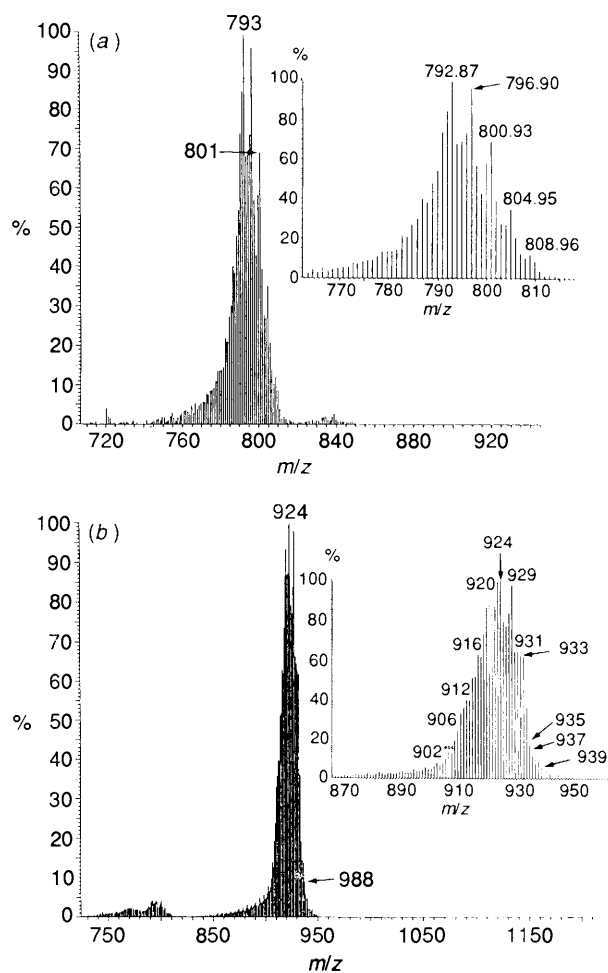


Fig. 5 Mass spectrum (EI conditions) for: (a) the product of reduction of [60]fullerene by Zn-conc. DCl in [$^2\text{H}_6$]benzene; (b) the product of reduction of [70]fullerene by Zn-conc. DCl in [$^2\text{H}_8$]toluene

without agitation survive largely unchanged for 2 weeks, and solid samples are more stable than those kept in solution. The instability towards light is reminiscent of the behaviour of C_{60}H_2 in the presence of C_{60} reported by Henderson and Cahill.¹⁸ A toluene solution of $\text{C}_{60}\text{H}_{36}$ through which air had been bubbled for 48 h (in the light) showed (after removal of solvent and standing in air for 24 h) total degradation to a water-soluble product, whose IR spectrum was similar to that of fullerenols.¹⁴

Reaction with carbon disulfide

Solutions of $\text{C}_{60}\text{H}_{36}$ in CS_2 produced a black precipitate within 5–10 mins in air, and within 30 min under nitrogen. The filtrate from this gave a mass spectrum indistinguishable from the original $\text{C}_{60}\text{H}_{36}$. The black residue gave the spectrum shown in Fig. 9, from which it is evident that substantial decomposition to $\text{C}_{60}\text{H}_{18}$ has occurred.

Formation of trimethylene adducts

$\text{C}_{60}\text{H}_{36}$ is resistant towards further reduction with hydrogen in the presence of a catalyst, such as rhodium on alumina. However, in these experiments we obtained evidence for the increased formation of species first noted in the products of the initial Zn-HCl reductions; these had $m/z = 780, 798$ and 840 . The latter was particularly puzzling since our initial material was entirely free of [70]fullerene.

Thus reduction of $\text{C}_{60}\text{H}_{36}$ with H_2 -Rh-alumina in toluene over 10 days gave mass spectra such as that shown in Fig. 10(a).

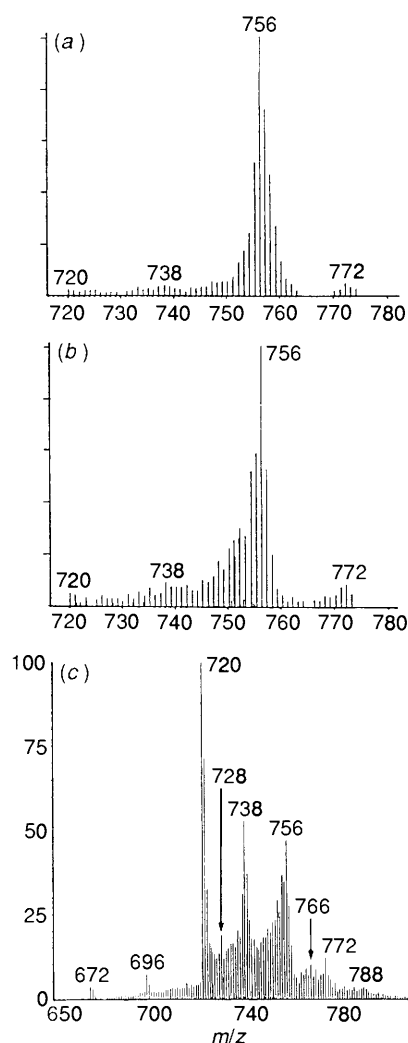


Fig. 6 Mass spectrum (EI conditions, DCl probe 650 °C) for $\text{C}_{60}\text{H}_{36}$ (a) taken initially; (b) taken 9 min later; (c) taken ca. 1 h later

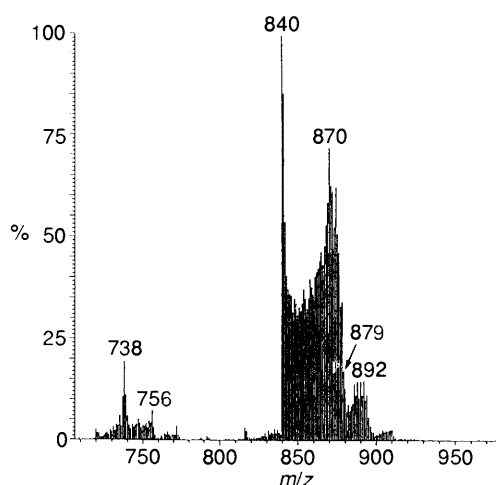


Fig. 7 Mass spectrum (EI conditions, DCl probe 650 °C) for $\text{C}_{70}\text{H}_{36/38}$ taken after 2 min on the probe

Three moieties each of 42 amu have added, which can reasonably be attributed only to $(\text{CH}_2)_3$ units. The origin of these species is unclear, but possibly they arise by abstraction from the methylcyclohexane produced from the solvent toluene during the reduction. In order to test this possibility, the reduction was repeated using [$^2\text{H}_6$]benzene as the solvent, since here the mass increments would be 45 amu. Not only did

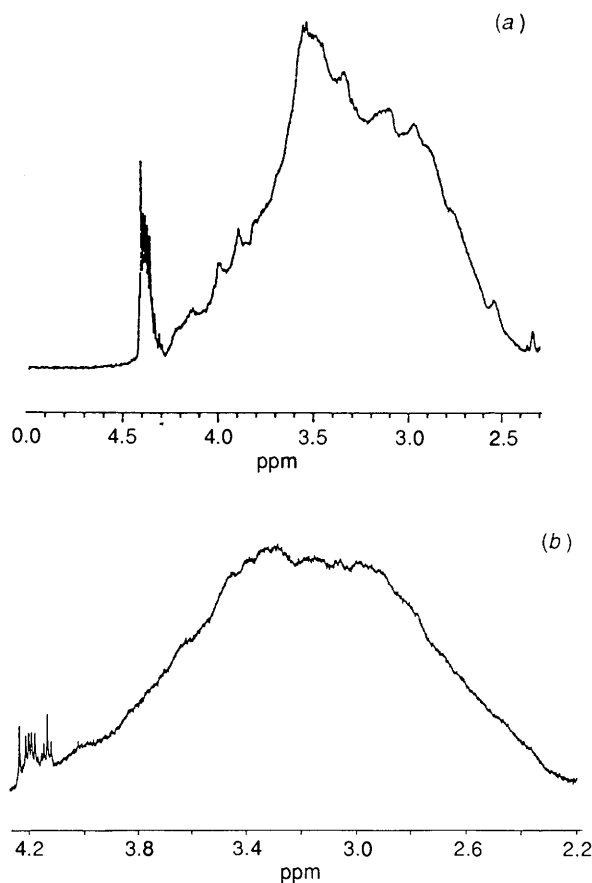


Fig. 8 ^1H NMR spectra for: (a) $\text{C}_{60}\text{H}_{36}$; (b) $\text{C}_{70}\text{H}_{36/38}$

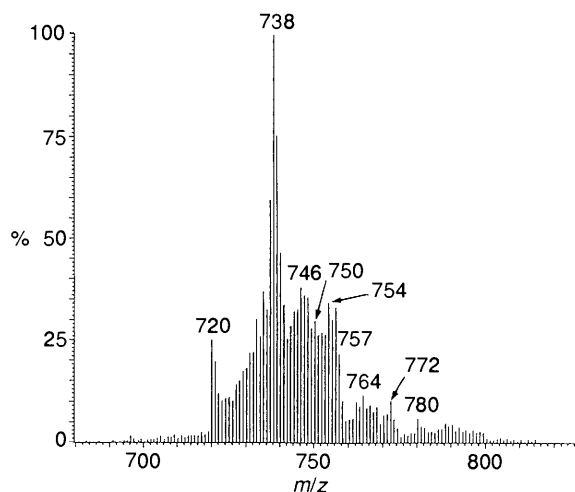


Fig. 9 Mass spectrum (EI conditions) of the residue from the reaction of $\text{C}_{60}\text{H}_{36}$ with CS_2

we not find any trimethylene addition in this case, but most surprisingly, the hydrogenated fullerene underwent partial hydrogen exchange with the solvent, giving a main peak at 759 amu, and a cluster of secondary peaks centred at 788–790 amu. We hope to investigate this phenomenon further.

Significantly, the species of $m/z = 780$ was observed only in samples in which $\text{C}_{60}\text{H}_{18}$ was also present [Fig. 10(b)], a result attributable to addition of $(\text{CH}_2)_3$ to $\text{C}_{60}\text{H}_{18}$. Previously we found the parent fullerenes to abstract $(\text{CH}_2)_3$ units readily from THF (tetrahydrofuran),¹⁹ but the origin of the tendency for trimethylene addition is unclear since the hydrogenated derivatives appear now to behave similarly. Particularly

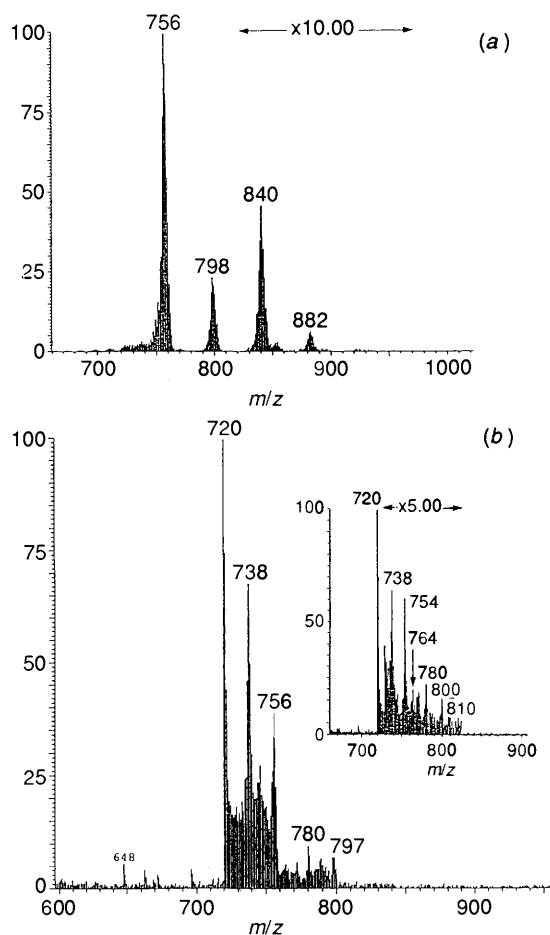


Fig. 10 (a), (b) Mass spectra (EI conditions) of $\text{C}_{60}\text{H}_{18/36}$ showing the presence of trimethylene adducts

puzzling is the apparent ease with which the reduced cage can acquire up to three trimethylene groups, yet is fairly resistant to further reduction.

Similar addition also occurs with hydrogenated [70]fullerene, as is evident from Fig. 4(b) which shows the addition of one and two trimethylene groups, giving $m/z = 918$ and 960 , respectively.

Hydrogen exchange

Hydrogenated fullerenes are believed to be acidic, e.g., $\text{C}_{60}\text{HBU}'$ was found to have a $\text{p}K_a$ of 5.7,²⁰ whilst $\text{C}_{60}\text{Hmorpholine}$ underwent exchange with MeOD .²¹ The acidity would be expected to decrease as the number of hydrogens attached to the cage increases as this would reduce the overall electron-withdrawing effect. This conjecture is confirmed by the shifts for the ^1H NMR spectra of the hydrogenated fullerenes, which lie further upfield the more hydrogens are attached to the cage; they are also more upfield for [70]fullerene derivatives since [70]fullerene is overall less electron-withdrawing than [60]fullerene.¹² Moreover, the locations of the NMR spectra for the derivatives described in this paper are considerably further upfield, suggesting that the hydrogen acidities are markedly reduced. This is confirmed by our finding that toluene solutions of $\text{C}_{60}\text{H}_{36}$ showed no change in the mass spectrum after being shaken with D_2O either alone or in the presence of either aq. sodium hydrogen carbonate or aq. sodium hydroxide (in the absence of light). Very recently, exchange of the cage-attached proton in methyl 1,2-dihydro[60]fullerenecarboxylate has been reported to be unexpectedly slow, possibly due to heterogeneity in this case.²²

The UV spectrum of C₇₀H₃₆

The UV spectrum of C₇₀H₃₆ shows a broad band centred at 275 nm and a pronounced shoulder at 223.5 nm. These features resemble very closely those for naphthalene λ_{\max}/nm 221 and 258–286, and are consistent with an aromatic structure proposed for this molecule, in which naphthalene moieties are present.¹⁷

Acknowledgements

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