

Quasicrystals as part of an unusual species of intermetallic phases

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Developments in the field of quasicrystallography are reviewed, particularly in the light of recent high-resolution X-ray diffraction data on quasicrystals. The possibility of most, if not all, icosahedral and decagonal phases forming part of a rather unusual species of crystalline intermetallic compounds is explored. An analytical study of available data on binary and ternary aluminium-transition metal alloys leads to the novel concept of *Icosahedral Cluster Compounds*, distinct from both stoichiometric chemical and Hume-Rothery electron compounds. These new intermetallic phases are shown to nucleate from ordered 13-atom icosahedral clusters and to grow in different ways, depending on their transition metal content.

MUKHOPHADYAY and Sinha¹ have reported on the Sixth International Conference on Quasicrystals held at Tokyo, Japan. Their brief coverage of 19 technical sessions, 45 oral presentations and 145 poster papers tends to paint, in the present author's view, a rather rosy and far from realistic picture of the present quasicrystal scenario. Convincing answers have not yet been given to the following three fundamental questions concerning quasicrystals, even though they have been raised on and off during the last decade:

- Where exactly are the atoms located in *three dimensions* in a quasicrystal?
- How do quasicrystals *nucleate and grow* from the liquid state?
- Why do quasicrystals occur at some *specific* and rather *unusual* compositions?

The 'fascinating discovery² of quasicrystals in 1984' and the resulting 'paradigm shift in the solid state' are too well-known to be elaborated here. In fact, they have been dealt with exhaustively in dozens of conferences around the world and referred to appreciatively in thousands of papers in diverse international journals. It is also a fact that all along, some scientists³⁻¹², among them Linus Pauling and the present author in particular, have taken the stand that the quasicrystal phenomenon, although somewhat baffling with its five-fold diffraction effects, may well be understood and explained within the broad framework of classical crystallography. Accordingly, they have deliberately chosen to tread the time-honoured and beaten path of traditional crystallographers. Even among the votaries of quasicrystallography, some have raised probing questions or

highlighted worrying facts, that seem to cast doubts on the soundness of the quasicrystallography edifice¹³⁻²⁰. Meticulous experimental work¹⁶⁻²⁰ during the last five years seems to suggest that there are really no quasicrystals at room temperatures, even though they may occur at higher temperatures.

According to many X-ray specialists active in this field, Debye-Scherrer or 'low-resolution' X-ray diffraction is not of much use in throwing light on unusual and complex twinning/hypertwinning problems one may encounter in quasicrystals. State of the art is single crystal or 'high-resolution' X-ray diffraction using synchrotron radiation, which is unfortunately beyond the reach of most laboratories in the world. However, this expensive and sophisticated technique has been recently put to good use by a few groups in Europe, notably by Denoyer and her co-workers at Paris and by Steurer's team in Zürich. A distinction has been made in publications of these groups among quasicrystals, approximants and rational approximants. One fundamental issue that recent intensive researches have thrown up relates to the limits of our ability to distinguish clearly through experiment between genuine quasicrystals and multiply-twinned, nanodomain crystalline approximants, generally with rather large unit cells. In short, as admitted by many quasicrystallographers, it has now become difficult, if not impossible, to establish whether a particular specimen is a true quasicrystal or not.

Although icosahedral and decagonal quasicrystals are based on quite a few metals like aluminium, gallium, titanium, vanadium and magnesium, more than 90% of the publications in this field relate to binary, ternary and quaternary alloys based on just one metal, viz. aluminium (Al). Thus a critical survey of all known complex crystalline, quasicrystalline and rational approximant phases may be considered as a good starting point for a possible unified approach to solidification of different types of unusual phases, stable as well as metastable, from Al-alloy melts. What strikes a keen observer of this vast field in the first instance is that the compositions of the diverse phases do not generally correspond to what one would normally expect from chemical or stoichiometric compounds or even electron compounds, the so-called Hume-Rothery phases. It looks as though we are concerned here with a new species of intermetallic phases following rules which are yet to be clearly perceived and understood.

In a seminal paper on eutectics (low-melting alloys) in binary metallic systems, Hume-Rothery and Anderson²¹ showed in 1960 that eutectic composition distribution curves display a series of peaks at 7-8 atomic% intervals, i.e. in atomic fractions at multiples of 1/13. This study was not undertaken with special reference to aluminium alloys, but covered all binary phase diagrams, known at that time, i.e. nearly 40 years ago. However, it

brought out an important fact, which seems to be of immense significance today in our efforts to probe and understand the connecting link between complex crystals, quasi-crystals and their crystalline approximants in aluminium alloys. It was found that the melts of a large number of binary alloy systems AB, where the atoms of B are smaller than those of A, tended to acquire thermodynamic stability and to lead therefore to eutectics at 7–8 at.% intervals. Thus, with specific reference to aluminium alloys containing transition metals (M) whose atoms are smaller than those of aluminium, stabilization of alloy metals may be expected to occur at compositions represented by formulae like $Al_{12}M_1$, $Al_{11}M_2$, $Al_{10}M_3$, etc. The above-referred authors have explained that the stabilization of alloys melts at such unusual compositions can be easily understood in terms of ordering, i.e. preferential occupation of atomic sites by transition metal atoms without coming close to each other, within the 13-atom icosahedral clusters (1–5–1–5–1), the so-called Frank units, or in the pentagonal chains formed through vertex joining of the Frank units. Thus an $Al_{12}M_1$ cluster can be visualized by the ordered arrangement (1Al–5Al–1M–5Al–1Al), $Al_{11}M_2$ by (1Al–4Al, 1M–1Al–1M, 4Al–1Al), $Al_{10}M_3$ by (1M–5Al–1M–5Al–1M) and so on. The possibilities for ordering in pentagonal chains are numerous and may well present unusual complexities in long chains, i.e. (1–5–1–5–1–5...1–5–1).

Surveying the vast field of aluminium alloy phases today, one is struck by the fact that the compositions and formulae of solid phases present a somewhat confusing and incoherent picture, mainly because of paucity of accurate and reliable data on compositions and atomic arrangements in the unit cells proposed for a large number of aluminium-rich phases. Even with regard to the aluminium-rich Al–Fe crystalline phase, which has been under study for over a century, there are still a few uncertainties. The number of atoms in the monoclinic unit cell has been differently estimated as 100 or 102, as deduced from the given formula, Al_3Fe or $Al_{13}Fe_4$ (4×25 or 17×6), and the composition narrowed down to around 23 at.% Fe—by no means a meaningful percentage! In the light of observations in foregoing paragraphs and after a study of related phases in binary and ternary alloys of aluminium, the present author is inclined to settle for the formula $Al_{10}Fe_3$ with 23.1 at.% Fe and the likely number of atoms in the unit cell as 104 (13×8).

It is also significant that the number of atoms going with the formulae assigned by different investigators to diverse aluminium-rich intermetallic crystalline phases, mostly reported during the past four decades, works out to 13. Thus one can locate the following example²² with cubic, tetragonal, hexagonal, orthorhombic or monoclinic symmetry, but with all the concerned unit cells containing 26, 52 or 104 atoms (i.e. multiples of 13) in them:

1. $Al_{12}W$, $Al_{12}Mo$, $Al_{12}Cr$, $Al_{12}Mn$, $Al_{12}Re$ and $Al_{12}Tc$.
2. $Al_{11}Cr_2$, $Al_{11}V_2$ (also referred at times at $Al_{45}Cr_7$ and $Al_{45}V_7$).
3. $Al_{10}Fe_3$ (as above), $Al_{10}Co_3$, $Al_{10}Ru_3$, $Al_{10}Rh_3$, $Al_{10}Mn_3$, $Al_{10}Os_3$, $Al_{10}Fe_2Ce$, $Al_{20}Fe_5Ce$, $Al_{10}Cr_2Ca$, $Al_{20}Cu_2La$.
4. Al_9Mn_3Si , $Al_9Mn_2Si_2$, Al_9Fe_3Sm .
5. Al_8Fe_4Ca , Al_8Cu_4Ca , Al_8Mn_4Ca and dozens with rare earth (RE) elements, with compositions $Al_8Fe_4(RE)_1$, $Al_8Cr_4(RE)_1$ and $Al_8Cu_4(RE)_1$.

As will be shown later, the compositions of quasicrystalline phases also follow such a pattern.

Shortly after the advent of the quasicrystal era, early in 1986, Rao and Kulkarni²³ drew attention to the often overlooked fact that aluminium is a third group element next to boron, an element notorious for its bewildering and unusual allotropy, characterized by numerous closely related atomic arrangements with varying structure types, all of them built with the B_{12} icosahedral 'brick'. (The icosahedra contain 12 atoms only with the central, 13th atom, missing. Such a 12-atom icosahedron is designated 'empty', while 13-atom icosahedron is referred to as 'filled', in the scientific literature.) The unit cell of a particular modification of boron has been reported to contain no less than 1708 atoms in an 'ordered labyrinth' of B_{12} icosahedra! In case of aluminium, as rightly observed by these authors, addition of transition metals in specific proportions and with appropriate smaller atomic size is likely 'to revive its latent tendency' to form icosahedral structures. In fact, their energy calculations using extended Hückel, EH, method with Slater type atomic basis sets (using 3s, 3p, orbitals of Al and 3d, 4s, 4p orbitals of Mn) showed that the $Al_{12}Mn$ icosahedron is energetically *more stable* than the $Al_{12}Mn$ cubo-octahedron, particularly in the molten alloys.

A more recent and even more significant development concerns studies by Gong and Kumar²⁴ on the electronic structure and relative stability of icosahedral Al-transition metal (Al–M) clusters, particularly $Al_{12}M$, where M represents the generally smaller transition metal atom. Based on density functional theory within the local spin density approximation, their calculations predict large binding energies for clusters with atoms of M metal coming from elements in the *middle* of the d series and occupying the body centre positions in the icosahedron—in general agreement with observations on formation of quasicrystals. Their computations also show interesting trends in sp-d hybridization in a d series, which not only provide for the interaction between M atoms in the icosahedral phases, but also lead to a contraction of the Al–M bond, resulting in better icosahedral packing, such as in the cases of Fe, Co, Ru, Pd and Cu.

Yet another significant development relates to observations of 'channels' apparently built of regular icosahedra.

hedra²⁵ or distorted icosahedra²⁶ in crystalline phases like the Al-Fe compound referred to above and also in quasicrystalline phases like those of Al-Mn system. The different periodicities of around 4, 8, 12, 16 Å, etc. in decagonal quasicrystals have, in fact, been explained on the basis of complex ordering in such channels²⁷. Considering the pentagons normally rotated 36° to each other, let us call them A and B, in individual icosahedral clusters and in pentagonal chains along the vertex line or chain axis, providing an ABABAB-decagonal channel, repeated rotation of pentagonal pairs by +18° and -18° respectively in the liquid state will convert the same to an AAAA-pentagonal channel. Regular or random rotation of the pentagons thus paves the way for an enormous number of regular or faulted sequences involving A and B positions and thus generate numerous 'polytypes' with very little energy differences between them, as has actually been observed in rational approximants to quasicrystalline phases.

The present author summed up the ideas and suggestions gradually developed by him over nearly a decade in his 1994 review article¹¹ dedicated to the memory of Professor Linus Pauling, in the following words: 'The golden thread running through the numerous aluminium-transition metal phases, viz. metallic glasses, icosahedral and decagonal quasicrystalline phases, rational crystalline approximants, Frank-Kasper and similar complex phases, can now be identified as the icosahedral atomic cluster present in the Al-TM alloy melts. Starting from such clusters, the crystalline phases form through homogeneous nucleation in two ways, one leading to orthorhombic, hexagonal and rhombohedral structures, that have a propensity for multiple twinning and the other leading to complex cubic structures, while the metallic glasses form by the random freezing-in of individual clusters and vertex-joined pentagonal chains of atoms from the liquid state'.

In the light of all the diverse facts and figures presented above, the golden thread may now be defined with a minor modification and with perhaps greater confidence than before as ordered 13-atom icosahedral clusters either as such, or as vertex-joined pentagonal chains of atoms. The latter chains made up of 13-atom clusters will contain $(12n+1)$ atoms, where n is the number of clusters, i.e. 25, 37, 49, 61, 73, 85, 97, etc. atoms. Summing up, we are concerned here with a new species of intermetallic phases that may be designated as Icosahedral Cluster Compounds. These new phases may be expected to form in aluminium alloys when certain conditions are fulfilled, viz.

1. Stabilization of the icosahedral clusters in the liquid state by either transition metal atoms only or a combination of transition and non-transition metal atoms, of appropriate sizes with ordering, making a further contribution to stability.

2. Strong electronic interactions between aluminium atoms and transition metal atoms leading to $sp-d$ hybridization in the d series, large binding energies and contraction of the Al-M bonds.

3. Compositions of these compounds decided not by stoichiometry as in the case of chemical compounds or electron concentrations per atom as in the case of electron compounds (i.e. Hume-Rothery phases); but by the nature of ordering of M-atoms in the clusters/hyperatoms based on simple and straight-forward considerations.

4. Nucleation and growth of the solid phases from hyperatoms in the liquid state in three distinct ways, depending on the nature and content of the solute atoms, as also electronic structure and interactions between atoms: (a) The 13-atom clusters or basic hyperatoms do not come close enough to share atoms with neighbours, but align themselves to give rise to a cubic or tetragonal structure (e.g. bcc $Al_{12}W$, $Al_{12}Mo$, etc. with 26 atoms in the unit cell (Figure 1a). (b) The 13-atom clusters or basic hyperatoms join and weld themselves with atom sharing in all three directions, first to form a basic orthorhombic cell, which is but a distorted icosahedron retaining some of its original geometry, and then to grow into different, generally complex and often multiply twinned super-structures with orthorhombic or related rhombohedral/hexagonal/monoclinic symmetries (e.g. crystalline phases like $Al_{10}Fe_3$, $Al_{10}Co_3$, etc. and binary quasicrystalline phases like $Al_{85}Cr_{15}$, $Al_{80}Mn_{20}$ icosahedral and Al_3Ir , $Al_{75}Mn_{25}$ decagonal phases). (c) Bigger clusters grow around the 13-atom basic hyper atoms (e.g., Mackay icosahedra, Pauling triacontahedra, etc. (Figure 1) and these hierarchical hyperatoms build different crystal structures, two-cluster bcc, four-cluster fcc or eight-cluster β -W type (e.g., Frank-Kasper crystalline phases and related quasicrystalline phases).

It follows from the above specific conditions that the proposed Icosahedral Cluster Compounds are different from and more complex than chemical compounds or Hume-Rothery phases.

A detailed analysis of all the available data on aluminium alloys on the basis of the above postulates will take considerable time and effort, but the following preliminary analysis is presented from data in three reviews^{19,22,28}, even though the measurements of compositions are often tentative and inaccurate, particularly in case of quasicrystals:

Binary alloys (Al_1M_1). The following are likely percentages of M atoms on ordering in 13-atom clusters, as also in pentagonal chains tending to $12N$ atoms, with N being a large enough integer:

$Al_{12}M_1-Al_{12}M_2$	7.7%-8.3%
$Al_{21}M_3$	12.5%
$Al_{11}M_2-Al_{30}M_4$	15.4%-16.7%

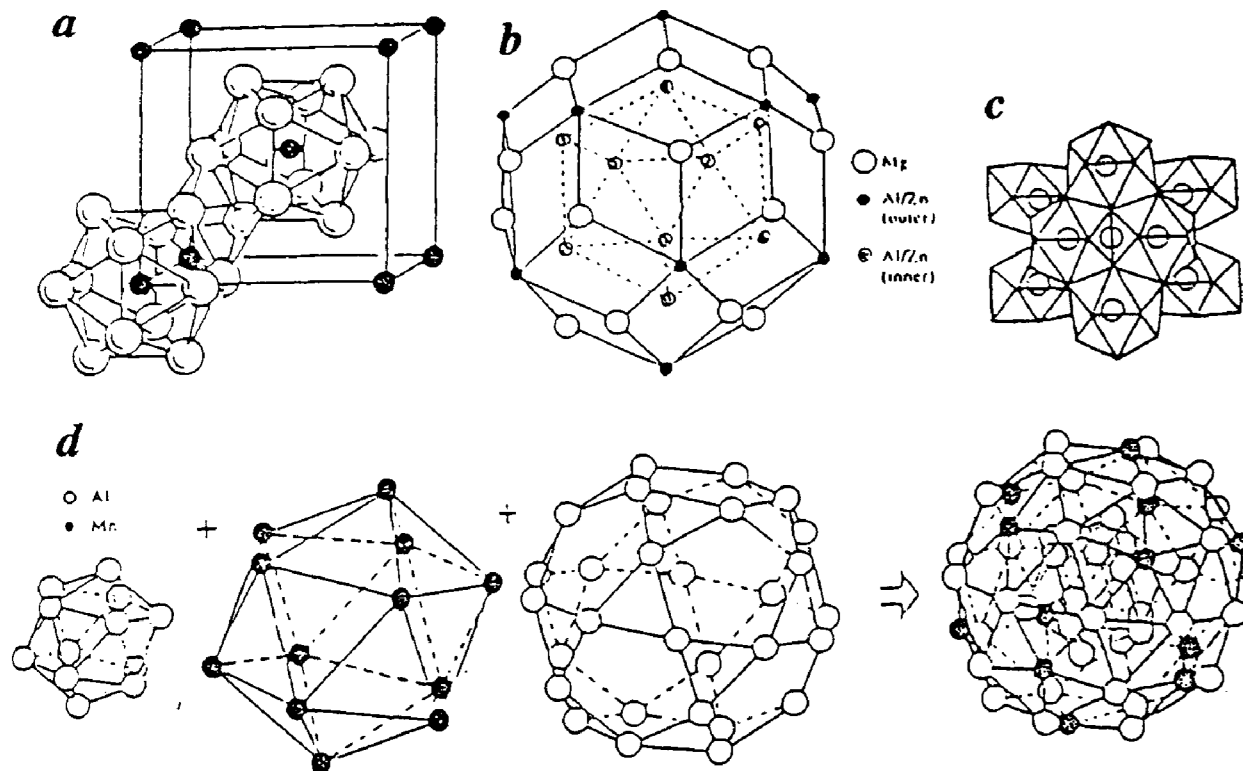
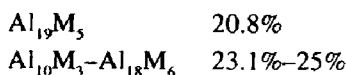


Figure 1. The 13-atom icosahedral cluster and the formation of bigger clusters around it. *a*, Unit cell of Al_{12}M (where $\text{M}=\text{W}, \text{Co}, \text{Cr}$, or Tc) with the basic cluster at origin and body centre. *b*, Pauling tricontahedron with 81 atoms, the cluster in $(\text{Al}, \text{Zn})_{49}\text{Mg}_{32}$ crystalline phase, as also in icosahedral Al-Mg-Zn quasicrystalline phase; *c*, Same cluster as in *b*, but with 12 basic clusters sharing edges with distortion and atom sharing; *d*, Mackay icosahedron with 54 atoms, the cluster in $\alpha\text{-AlMnSi}$ crystalline phase, as also in icosahedral Al-Mn-Si quasicrystalline phase.



In binary Al alloys one cannot have more than 25 at.% of M atoms because of the inherent restriction in ordering, viz. M atoms cannot be near neighbours, but will have to be in touch with or surrounded by Al atoms.

Examples:

1. Quasicrystalline phases

- Decagonal Al_5Zr , Al_5Pd , Al_5Pt , Al_5Ru and Al_5Os – 16.7%
- Icosahedral $\text{Al}_{85}\text{Cr}_{15}$ –15.0%; $\text{Al}_{80}\text{Mn}_{20}$ –20.0%; $\text{Al}_{79}\text{Rh}_{21}$ –21.0%
- Decagonal $\text{Al}_{80}\text{Fe}_{20}$ and $\text{Al}_{80}\text{Ni}_{20}$ –20.0%; $\text{Al}_{75}\text{Mn}_{25}$ –25.0%.

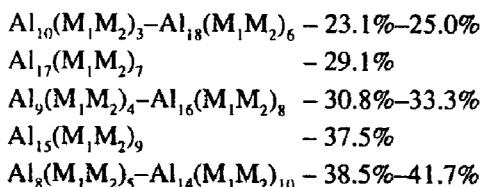
It is an important fact that no quasicrystal has been produced in binary aluminium alloys with more than 25 at.% of M, thus lending support to the above approach based on the ordered 13-atom clusters.

2. Crystalline phases

- Al_{12}W , Al_{12}Mo , Al_{12}Cr , Al_{12}Mn , Al_{12}Re and Al_{12}Te – 7.7%

- $\text{Al}_{11}\text{Cr}_2$ and Al_{11}V_2 – 15.4%
- $\text{Al}_{10}\text{Fe}_3$, $\text{Al}_{10}\text{Co}_3$, $\text{Al}_{10}\text{Ru}_3$, $\text{Al}_{10}\text{Rh}_3$, $\text{Al}_{10}\text{Mn}_3$ and $\text{Al}_{10}\text{Os}_3$ – 23.1%

Ternary alloys ($\text{Al}_x[\text{M}_1\text{M}_2]_y$). M_1 and M_2 together can account for more than 25 at.% because of the further flexibility in M_1M_2 contacts in the ordering patterns. Thus the following are likely percentages, starting from $\text{Al}_{10}(\text{M}_1\text{M}_2)_3$:



Examples:

1. Quasicrystalline phases

- Icosahedral $\text{Al}_{75}\text{Pd}_{15}\text{Co}_{10}$ – 25.0%
 $\text{Al}_{71}\text{Pd}_{20}\text{Re}_9$ – 29.0%
 $\text{Al}_{70.5}\text{Pd}_{22.5}\text{Mn}_8$ – 30.5%
 $\text{Al}_{62.3}\text{Cu}_{24.9}\text{Fe}_{12.8}$ – 37.7%
 $\text{Al}_{62}\text{Cr}_{19}\text{Si}_{19}$ – 38.0%

- Decagonal $\text{Al}_{75}\text{Ni}_{15}\text{Co}_{10}$ – 25.0%
- $\text{Al}_{71}\text{Ni}_{24}\text{Fe}_5$ – 29.0%
- $\text{Al}_{70.5}\text{Mn}_{16.5}\text{Pd}_{13}$ – 29.5%
- $\text{Al}_{70}\text{Ni}_{15}\text{Rh}_{15}$ – 30.0%
- $\text{Al}_{62}\text{Cu}_{17.5}\text{Cu}_{17.5}\text{Si}_3$ – 38.0%

2. Crystalline phases

- $\text{Al}_{10}\text{Fe}_2\text{Ce}$, $\text{Al}_{20}\text{Fe}_5\text{Ce}$, $\text{Al}_{10}\text{Cr}_2\text{Ca}$ and $\text{Al}_{10}\text{Cu}_2\text{La}$ – 23.1%
- $\text{Al}_9\text{Mn}_3\text{Si}$, $\text{Al}_9\text{Mn}_2\text{Si}_2$, $\text{Al}_9\text{Mn}_3\text{Ca}$ and $\text{Al}_9\text{Fe}_3\text{Sm}$ – 30.8%
- $\text{Al}_8\text{Fe}_4\text{Ca}$, $\text{Al}_8\text{Cu}_4\text{Ca}$, $\text{Al}_8\text{Mn}_4\text{Ca}$ and many dozen others with rare earth elements – 38.5%

In conclusion, it is hoped that the above facts, figures and postulates, particularly the encouraging preliminary analysis of compositions, will be considered adequate for justification for further work by scientists in this field to explore systematically and understand clearly the special features of the proposed new species of Icosahedral Cluster Compounds, not only in aluminium alloys, but in other related alloy systems.

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Modification of genotoxicity of radiation by post-irradiation treatment with caffeine

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The effect of caffeine during post-irradiation growth period on survival, gene conversion, back mutation, aberrant colony formation (ACF) and mitotic crossing over (MCO) was investigated in the diploid yeast strain *Saccharomyces cerevisiae* D7. The presence of caffeine in growth media reduced the radiation-induced cell killing and the frequency of all the genetic end points except MCO. The increase in MCO frequency might be related to the enhanced cell survival brought about by caffeine. It is suggested that post-irradiation division delay introduced by caffeine may alter the post-irradiation repair processes and may help the process of recovery by error-free repair pathways.

CAFFEINE is an environmental chemical to which people are exposed through food items, beverages and medicines¹. A number of workers have reported a mutagenic activity of caffeine in bacteria, yeast and cultured mammalian cells². In addition, caffeine has been shown to modify the mutagenic activity of several chemical mutagens, UV and ionizing radiations in these systems^{3–8}. Caffeine also modifies carcinogenic effects of chemicals⁹. In yeast, caffeine has been reported to modify the lethal, mutagenic and recombinagenic effects of UV and *N*-methyl-*N'*-nitro-*N*-nitrosoguanidine (MNNG)^{10–12}. Caffeine enhances cytotoxicity of non-ionizing radiation in mammalian cells^{13,14}. With ionizing radiation, caffeine has been shown to exert both protecting and sensitizing effect. When present during irradiation, caffeine offers protection in cell culture systems¹⁵, plant seeds^{16,17}, and bacterial spores^{18,19}. Caffeine also exhibits radioprotection

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