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Prompt gamma neutron activation analysis (PGAA): recent developments and applications

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Abstract Prompt gamma neutron activation analysis has become an important part of the analytical toolkit, practiced at several research reactors worldwide. An extensive review of the physics, engineering, and applications of the technique was published in 2004: the present work gives an overview and survey of the literature in the succeeding dozen years.

Keywords Neutron capture · Prompt gamma activation analysis · Review · Neutron beam

Introduction

When a neutron is captured by a nucleus, its binding energy is released rapidly in the form of gamma rays characteristic of the compound nucleus. If these gammas are detected and analyzed by a high-resolution gamma spectrometer, the elemental and isotopic composition of the target material can be determined: this is prompt gamma activation analysis (PGAA). All elements except He emit capture gamma rays that can be detected for quantification, but in practice their measurability depends on the matrix in which the element of interest occurs. As many as thirty elements have be measured in a simultaneously in a single material by this essentially nondestructive technique. PGAA shares the metrological qualities of neutron activation analysis: independence of chemistry and traceability to the International System of Measurement [1, 2]. The first applications used thermal neutron beams at research reactors [3–7]. Improved sensitivity is obtained by using guided beams of thermal [8] or cold neutrons [9–16]. The following Fig. 1 gives a broad-brush picture of detection limits one such system.

Although industrial PGAA measurements are widely employed using radioisotopic neutron sources (^{252}Cf, ^{241}Am-Be, ^{238}Pu-Be) or neutron generators (using ^{2}H(d,n), ^{3}H(d,n), ^{7}Li(p,n), ^{3}H(p,n), or ^{9}Be(d,n) reactions) for the analysis of bulk materials [17, 18] such as coal and cement, explosives detection, and geological borehole mapping [19], these applications are largely outside the scope of the present review, which focuses on laboratory measurements with neutron beams. The early history of PGAA with both kinds of neutron sources have been summarized in a 1995 book [20].

In 2004 the *Handbook of Prompt Gamma Activation Analysis with Neutron Beams* [21] was published, containing detailed information on the physics, practice, and applications of the method, as well as the results of recent careful and extensive measurements of the capture gamma-ray spectra of the elements, in print and on CD-ROM. The present review focuses on the science and applications since that *Handbook* was published. After a brief introduction, recent developments in PGAA facilities will be outlined, followed by a discussion of recent improvements and extensions in the method. New and improved nuclear data obtained via neutron capture will be surveyed, followed by an overview of the numerous and varied insights that have been made possible by application of PGAA to problems in materials science, geology, archaeometry, biology, and medicine. This review surveys the English-language journal literature between 1995 and mid-2017; inevitably, some applications appearing in conference

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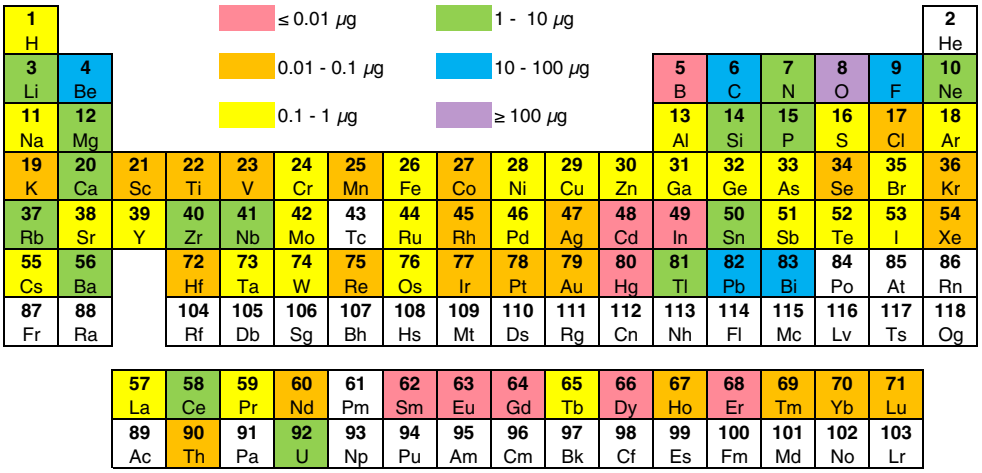

Fig. 1 Matrix- and background-free detection limits for PGAA in the NIST cold neutron beam, assuming a 24-hour measurement and $u = 10\%$ precision from counting statistics. The numerical values are indicative of the technique, but depend strongly on the characteristics of the particular measurement system employed. Relative element-to-element sensitivities scale well from one system to another

proceedings or specialized journals peripheral to the nuclear field have been missed.

An important resource for the *Handbook* was an IAEA Coordinated Research Program; the published report of that CRP [22] gives much detail on the methods, and extensive tables of results on CD-ROM. The data are stored in the Evaluated Gamma-ray Activation File (EGAF) [23], and available on line at https://www-nds.iaea.org/pgaa. A detailed description of elemental analysis by the PGAA method with a full ISO-compliant computation of uncertainties was published in 2009 [24], and the method was reviewed in the *Encyclopedia of Analytical Chemistry* [25]. Emphasizing the physics, a 54-page review with 239 references was included in the second edition of the *Handbook of Nuclear Chemistry* in 2011 [26].

Facilities

Since the *Handbook* was published, a number of new PGAA facilities have been described, and existing installations upgraded. In Argentina a PGAA system optimized for ^{10}B measurements at the RA-3 reactor is under development [27], and in Brazil a facility at the IPR-R1 reactor is being designed [28]. Two facilities in China are in operation [29], one related to boron neutron capture therapy (BNCT) at the 30-kW In-Hospital Neutron Irradiator (IHNI) in Beijing [30], and another general-purpose instrument at the China Advanced Research Reactor [31, 32].

In Germany, PGAA is vigorously pursued at the FRM II reactor at the Technical University of Munich (TUM) in Garching (Heinz Maier-Leibnitz Zentrum—MLZ). The instrument was originally operated at the spallation source SINQ at Paul Scherrer Institut, Villingen, Switzerland until 2000 in a medium-flux cold neutron beam [33, 34]. The facility was redesigned at the University of Cologne, moved to Garching, and installed on a high-flux cold neutron beam at FRM II. The new instrument came into service in early 2007 [35–37]. The optimized instrument [38] features a focusing guide section with the highest flux (6.1×10^{10} cm^{-2} s^{-1} thermal equivalent) yet available for PGAA at the focal point. This flux is high enough for in-beam activation analysis with off-line gamma assay. A parallel beam with the flux of 2×10^{9} cm^{-2} s^{-1} is also available for standard PGAA applications. Great emphasis was laid on the reduction of the spectral background, using an optimized arrangement of shielding components and also with Compton suppression. Complementary neutron imaging can be done as well [39]. A specialized facility with a beam of fission-spectrum neutrons has been constructed at Garching, intended to study inelastic scattering and other fast-neutron reactions [40, 41].

The Centre for Energy Research of the Hungarian Academy of Sciences, a member of the Budapest Neutron Centre (BNC), is a pioneer in nuclear analysis and continues to advance the science and applications of PGAA [42] and other methods. The PGAA station began operation at the end of the 1990s as a thermal beam facility, with the flux of 2.3×10^{6} cm^{-2} s^{-1}. In spite of the low flux, the group made numerous developments in methodology: e.g., broad-range energy calibration [43], an improved spectroscopy data library [44], evaluation software [24, 45], and neutron spectrum modeling [46]. After a cold neutron source was installed the flux reached 10^{8} cm^{-2} s^{-1}, which enabled a wide range of applications and further developments [47, 48]. The facility now accommodates two PGAA instruments on the same beam (the second one with about three times less flux) which can be run practically independently. Both instruments use Compton-suppressed HPGe detectors, and a low-energy HPGe detector is also available for high-resolution gamma counting below 1–2 MeV [49]. With the combination of PGAA and neutron tomography (NT) for neutron-tomography driven PGAA, strongly collimating both the neutron beam and

detected gamma rays for prompt gamma activation imaging (PGAI) [47, 50–52] the facility has made possible a wealth of applications, which will be discussed in a later section. PGAA measurements at the ISIS Spallation Neutron Source in the United Kingdom have employed neutron energy selection to selectively enhance the signal from certain elements; the results have been compared with those from Budapest [53].

PGAA has been established at the Dhruva reactor at the Bhabha Atomic Research Center (BARC) in India to complement conventional neutron activation analysis [54]. Before the 2011 Fukushima accident shut it down, an imaginative PGAA application with multiple gamma-ray detectors was being developed at the JRR-3 M reactor in Japan [55]. A large number of applications of PGAA, to be discussed later, have been carried out at this reactor, and will doubtless continue when it is returned to service. Measurements employing PGAA in combination with time-of-flight elemental analysis are underway at the J-PARC pulsed-neutron source [56]. A diffracted beam is in service at the HANARO reactor in Korea [57]. Neutron beams at the Moroccan research reactor have been characterized by experiment and modeling [58]. A PGAA facility has been established at the Institute for Nuclear Research—Pitesti in Romania [59]. In Russia, a facility was built at the MEPhI reactor in Moscow, directed at neutron capture therapy [60]. Studies are underway in Thailand toward establishing PGAA at the Thai Institute of Nuclear Technology [61, 62].

PGAA facilities in the United States were reviewed in 2005 by Ünlü [63]. Since then, the cold-neutron instrument at NIST has been moved to the end station of a new beam with 10-fold higher flux and more flexible experimental space [64, 65]; the system is under continual development [66]. The older thermal-neutron PGAA instrument continues in service as well [67]. The characteristics of the two systems are complementary [68]: higher flux and lower background at the cold beam give greater sensitivity, whereas the analysis of highly scattering samples such as biological tissues and polymers generally uses the thermal beam, in which the complication of change of neutron temperature, and thereby change of cross section, is absent [69]. Reactor beams have been newly reshaped and used for PGAA at Ohio State [70] and Oregon State [71] Universities. The instrument at the University of Texas has been reconfigured for improved performance [72].

Methods and new science

An essential step in preparing a PGAA system for use is a careful and complete characterization of its performance, especially the neutron flux, elemental sensitivity, and background [47]. An important parameter is the neutron temperature, which is readily determined by comparing thick and thin samples [73]. The background components of the Garching [38], Texas [74], and Budapest [75] facilities have been described. Thermal and cold neutron beams at NIST were compared for the determination of C, N, and P in biological samples in which neutron scattering is important [76, 77]; accurate measurement required corrections that depended on the H and Cl contents of the materials.

These calibration measurements are increasingly being supplemented by Monte Carlo calculations [46, 64, 78, 79]. When a calculation is benchmarked against a physical measurement of the same configuration, the effect of changes in the shielding and other parameters can be predicted with confidence [80–82]. Modeling of the neutron beam [62, 83], the transmission of the neutrons and the resulting gamma rays within the sample [84], and the interaction of the gammas with the detector [85, 86] all aid in interpreting the resulting spectra in order to yield the most accurate and useful information on the sample composition.

Modeling calculations of neutron guides have proven to be essential in optimizing the signal/background ratio in PGAA instruments [64, 80, 87, 88]. Gamma radiation from the beam stop is a particular issue: if each neutron in a 10 cm^2 beam of 10^8 n/cm^2 s produces one gamma ray, the radiation source strength is 10^9 Bq, or 30 mCi. Stopping the beam in boron produces 0.94 gamma rays per neutron, but this 478 keV radiation is readily shielded, especially if the beam stop is located far from the detector. The reaction $^6Li(n,T)^4He$ is gamma-free, but the energetic tritons produce fast neutrons which degrade the detector and are difficult to shield. Helium-3 is perhaps the most nearly ideal [65], but it is expensive and requires a container. For some measurements (notably separating 20-ms ^{24m}Na from ^{10}B capture), it is useful to chop the beam in time to separate short-lived activation products from capture events [89, 90].

For most nuclides the cross section for slow-neutron capture is inversely proportional to the neutron velocity (the "1/v law") [91]. As a result the analytical sensitivity is better for cold neutrons than thermal. However, certain nuclides (mostly metallic elements) that show resonance peaks at super-thermal energies may be more selectively measured by using epithermal neutron beams [92–94]. Specially designed epithermal [79, 95] and fission-spectrum [41] neutron beams have been used. Still more selectivity is possible with neutrons from pulsed sources, using time of flight to select a narrow range of neutron energies to probe the sample: this is called neutron resonance analysis (NRCA), an emerging complementary

technique to PGAA [53, 94, 96]. Sufficiently energetic neutrons may lead to broadened gamma-ray peaks [97, 98].

Software is an essential component of a PGAA system to control the collection [99] and analysis [45, 100] of the gamma-ray spectra. The 478-keV peak from ^{10}B capture is a special case [101–103]. Since this gamma is emitted by the recoiling ^{7}Li product, the peak is heavily Doppler shifted by an amount that depends on the matrix; this dependence has been used to show the location of boron in seeds [101]. Several sources of random and systematic uncertainty beyond counting statistics need to be taken into account in a full calculation of the uncertainty of reported values [24, 104, 105]. These include fluence monitoring, neutron scattering, neutron self-shielding [106], and the parameters involved in standardization such as detector efficiency and standard masses [107]. Relative single-monitor standardization using the k_0 formalism [108, 109] is especially reliable for cold neutrons, where the cross sections generally follow the $1/v$ law.

A great deal of effort has gone into the characterization of gamma-ray detector performance. Efficiency as a function of energy is a perennial issue that has received great attention [110, 111]. Because of the high energy of capture gammas, optimum detector selection [49] and design are different than for conventional NAA. Compton suppression is highly beneficial in improving the peak/background ratio [51, 112]. Gamma-gamma coincidence with multiple detectors offers advantages in specificity, and increasingly in improved detection limits [113–115]. Digital pulse processing with multiple detectors opens new possibilities in data acquisition and analysis [116–118], especially when each detected event is time stamped [119]. With this approach, coincident events can be selectively examined with multiple coincidence criteria after data are acquired, without the need for tedious and sensitive timing adjustment required in analog logic [120–122].

Although the experimental conditions can often be arranged such that corrections for neutron spatial gradient, self-shielding, or dead time are small or negligible, certain samples require that these issues be considered explicitly. Very small samples call for the best combination of high flux, high detection efficiency, and low background [123]. Highly absorbing samples require that attention be paid to self-shielding [124, 125], and may benefit by using selected absorbers if low-energy gammas dominate the capture spectrum [126]. Samples larger than the neutron beam or thicker than a fraction of the neutron mean free path are especially sensitive to external [127] and internal absorption [128–131]. The involvement of imaging techniques can assist in carrying out the required corrections.

Although a characteristic and indeed desirable characteristic of PGAA is that the entire sample is analyzed, an inhomogeneous material can be characterized by repeated measurements with a neutron beam of limited size, which can ultimately be merged into an elemental image. An early application of this idea was published in 1987 [132, 133]. Because of higher flux, a focused neutron beam gives better analytical sensitivity than simple collimation [134]. A polycapillary bender-lens [135] has been used to form a cross-sectional image of a titanium lattice [136] by stepping a focused neutron beam across the sample and collecting data at 5 s per 1-mm^2 pixel. Because this was a converging beam, some depth resolution was obtained. Collimating both the neutron beam and the field of view of the gamma detector gives fine resolution of the image, at the cost of long measurement time. Extensive imaging measurements in combination with neutron tomography have been pursued in Budapest [52, 137, 138] and Garching [39, 139, 140], notably in connection with the characterization of archaeological and geological specimens. For example, at FRM-II a 3-dimensional image of a meteorite fragment was generated at a rate of 1 s per voxel with 0.4 mm resolution, after which 2-dimensional maps of the major elements were obtained at 1 h per 2 × 2.5 mm^2 pixel [139]. In later work, these methods were applied to an archaeological object as part of the ANCIENT CHARM project. In this work a 3-dimensional map of the major elements with 2-mm resolution gave insights into the history of a 6th-century fibula by entirely nondestructive means [140].

The combination of PGAA and instrumental neutron activation analysis (INAA) is routinely done in several laboratories, most often by separate irradiations in the beam and by sending samples into regions near the core. Both techniques employ neutron capture reactions independent of chemical state and use gamma spectrometry for quantitation. Both use similar technology, and the elements determined are complementary; in particular, the low-Z lighter elements have few delayed gamma-emitting capture products suitable for INAA [2]. Short-lived activation products are measurable immediately after the beam is turned off, by using a high-efficiency detector near the sample [38, 141]. A number of comparisons have been made between PGAA and other techniques. Using a focused beam from a polycapillary neutron lens, PGAA was used to map the chlorine distribution in silica at ~ 1 mg/g levels with 1 mm resolution; the results compared well with micro-x-ray fluorescence [134]. Chlorine was measured in 26 rock samples by both PGAA and isotope-dilution accelerator mass spectrometry at concentrations below 100 μg/g [142]. PGAA, inductively coupled plasma mass spectrometry, and quantitative neutron capture radiography were intercompared for the determination of boron in clinical tissue samples for BNCT [143].

Nuclear data

Capture gamma rays have been used for many years in studying nuclear structure, in particular the characterization of nuclear levels below the capture energy. Neutron capture cross sections are especially well suited to measurement with neutron beams [144, 145]. As previously mentioned, an IAEA Coordinated Research Program resulted in a comprehensive and consistent database of the relevant nuclear parameters for PGAA: gamma ray energies, abundances, and cross sections [22]. The spectroscopy tables and the spectrum atlas in the mentioned *Handbook* of PGAA [21] are being used for the chemical analyses, and the book is a useful source of additional nuclear data. The spectroscopy database is maintained in Garching.

Because many nuclear parameters are measured relative to a standard, characterization of these benchmarks is especially important [146]. A convenient calibration reaction is ^{14}N(n,γ), giving gamma-ray energies up to 10.8 MeV [147]. Because germanium gamma detectors are invariably used in PGAA with neutron beams, the capture spectrum of Ge is especially relevant [148–151]. Given the complexity of capture gamma spectra, small peaks in the spectrum of a matrix element may interfere with the determination of trace constituents.

Neutron beam facilities have been used extensively in measuring neutron capture cross sections and nuclear structure [144, 145, 152, 153]. The light nuclides ^{2}H, ^{6,7}Li, ^{9}Be, ^{10,11}B, ^{12,13}C, ^{14,15}N, and ^{16,17,18}O have been systematically studied [154–156], and k_0 factors and partial cross sections determined for B, N, Si, P, S, and Cl [157]. Other elements and nuclides that have been studied recently (listed in order of mass) are ^{22}Ne [158], ^{23}Na [159], ^{39,41}K [160], ^{56}Fe [161], ^{74,76}Ge [149, 150], $^{102-110}$Pd [162], ^{114}Cd [163], ^{115}In [164], ^{129}I, [165], ^{151,153}Eu [166], ^{155,157}Gd [167], ^{180}W [168, 169] and other W isotopes [170], ^{186}Re [171], ^{206}Pb [172], ^{209}Bi [173–175], ^{237}Np [176, 177], ^{241}Am [178–180], and ^{242}Np [177].

Directed toward PGAA specifically, k_0 factors have been measured for numerous elements [157, 181–186] in differing neutron spectra. A series of cross section measurements with fission spectrum neutrons is underway [40], as a part of the TANDEM project to study transuranic nuclides in more detail [187].

Applications

PGAA has proven valuable in a rich variety of applications. Recent examples will be described below in four broad groupings: materials science, biology and medicine, geoscience, and cultural studies.

Materials

Knowledge of the elemental composition of materials is essential in understanding their behavior. PGAA is being applied in a variety of contexts. The power of PGAA is employed in multielement analysis of substances such as rare earths [188], legacy radioactive materials [189] (even inside lead containers [190]), carbon nanotubes [191], fullerenes [192], $CaSO_4$ dosimeters [193], oxyanion materials [194], gas turbine alloys [195, 196], and cosmetics [197]. This technique's independence of chemical state is especially important in the certification of reference materials [198–204]. Explosives can be detected and identified by measuring characteristic H:C:N elemental ratios [205]. Neutron dosimeters have been developed that exploit measurement by PGAA of transformed isotopic ratios in materials exposed to high fluences [206].

Reported analyses of hydrogen [207, 208] and boron by PGAA are legion, because of the method's high sensitivity and specificity for these elements compared to other analytical techniques, and because of their technological importance. Hydrogen in metals is a continuing focus [66, 203, 208, 209], especially in titanium and steel. Concentrations below 1 mg/kg have been reported [210], but these measurements may not be reliable [211]. Storage media for a large-scale hydrogen economy have been studied [212, 213], notably a combinatorial measurement of thin hydride films of different composition [214]. PGAA of hydrogen has been used to monitor the chemistry of lithium-ion batteries [215], and to measure water in engineering composites [216] and building materials [217], and hydroxyl in oxide ceramic [218] and apatite [219]. Boron is readily determined due to its large cross section [220], and PGAA is thus a valuable tool in studying its complicated chemistry in classical chemical separations [200, 221]. As noted, PGAA is a preferred method for determining boron in reference materials [198, 201], and is used to measure B in steel [222], in foods [223], and in solution [224, 225]. Degradation of thin boron carbide films under neutron irradiation has been studied by PGAA [226].

Chemical elemental analysis is an essential component of physical structure studies of materials. PGAA has been used to characterize oxides [227], sulfides [228], complex nitrides [229–231], mullites [232, 233], and apatites for prosthetic applications [219]. Recent research in nanoparticle structure and reactions has benefited as well [234–236].

A promising new application for PGAA is the study of the mechanisms of chemical reactions, especially catalysis, in situ and in operando. The measurements were performed in a tube reactor with the neutron beam collimated on the catalyst bed, and the emitted gamma rays were detected.

The H-to-Pd (or to other catalyst metal) ratio was monitored as a function of experimental conditions, like temperature, pressure, gas composition and compared to the reaction product continuously monitored by a gas chromatograph. The H background of the gas mixture was subtracted based on the composition. The reaction mechanism was successfully interpreted based on these data [237–242]. Aging of electrochemical cells [243], nitrogen doping of oxide by combustion [244], reaction of isocyanates with acids [245], halogen production [246, 247], and hydrogenation [248] are some processes have been studied with the aid of PGAA. Diffusion studies of boron in diamond [249] and noble gases in porous media have been performed [250, 251].

Clinical studies, biology, and food

PGAA is employed for the analysis of foods for trace elements [143, 223, 252, 253] and total protein [254]. The major elements carbon, nitrogen, and phosphorus have been measured in plant tissue in environmental studies [255–257]. Boron is routinely measured in animal and human tissue in connection with BNCT for cancer [30, 258–260]. Gadolinium introduced in bone as a magnetic resonance agent has been quantified by PGAA [261].

Geochemistry and cosmochemistry

Numerous applications of PGAA to geology have been published, including multielement analysis of rocks [262–264], meteorites [265–267], coal and coal ash [268], sediments [269], impact glasses [270, 271], hydrothermal deposits [272], and mine tailings [273]. Hydrogen [274], boron [275–278], chlorine, [142, 275, 277], and rare earths [279] are of special interest in geochemical studies. Although not a laboratory measurement, it is noteworthy that the measurement of neutron capture gamma rays by spacecraft continues to be a powerful tool in the study of planetary surfaces [280].

Archaeometry and cultural heritage

The nondestructive nature of PGAA has been especially important in studies of archaeological and other materials in cultural history. Stone tools [281–283], ceramics and glass [284–291], iron [292–294], bronze and brass [295–301], coins [302, 303], and other metals and minerals [304, 305] have been studied. Position-sensitive PGAA and imaging methods are especially useful in this application area [286, 292, 300, 306–308], for example, the characterization of Ghiberti's doors in Florence [309]. An entire book chapter is devoted to PGAA imaging applications in archaeology [310].

Summary

The literature demonstrates a wide variety of improvements and applications of prompt gamma activation analysis in the dozen years since the publication of the definitive *Handbook*. We may expect continuing progress in the future.

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Glossary

BNCT	Boron neutron capture therapy
CD-ROM	Compact disk read-only memory
FRM II	Forschungs-Neutronenquelle Heinz Maier-Leibnitz
IAEA	International Atomic Energy Agency
INAA	Instrumental neutron activation analysis
MLZ	Heinz Maier-Leibnitz Zentrum
NAA	Neutron activation analysis
NIST	National Institute of Standards and Technology
NT	Neutron tomography
PGAA	Prompt-gamma activation analysis
PGAI	Prompt-gamma activation imaging
PGNAA	Prompt-gamma neutron activation analysis
SINQ	Swiss Spallation Neutron Source
TUM	Technical University of Munich

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