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Synthesis, Characterization and Mass Attenuation Coefficient of Green Graphene Sand Composite using Sr^{90} Beta Source

Richa Agrawal
Department of Physics
G. N. Khalsa College, Matunga, Mumbai, Maharashtra, India.
richa.agarwal@gnkhalsa.edu.in

Abstract: *Graphene is a flat monolayer of carbon atoms tightly packed into a two-dimensional honeycomb lattice and has completely conjugated sp^2 hybridized planar structure. Graphene and its derivatives havetremendous application potential. This paper consists of the synthesis of graphene sand composite (GSC)by a non-hazardous route. Samples of different thicknesses in disc shape were prepared and characterized by x-ray diffraction (XRD), infra-red (IR) spectroscopy, scanning electron microscopy (SEM)and Raman spectroscopy. Further the radioactive parameters such as linear attenuation co-efficient (LAC), mass attenuation coefficient (MAC), half layer value (HLV) and mean free path (MFP) have been evaluated from the experimental data by using Sr^{90} beta Source having energy of 2.27 MeV. Obtained results suggests that synthesised GSC can find applications in radioactive waste management, radiation attenuation/shielding and protective garments manufacturing..*

Keywords: Graphene sand composite, LAC, MAC, HLV, MFP,Raman spectroscopy

I. INTRODUCTION

Electromagnetic spectrum ranges from low energy radio waves to high energy gamma rays and have multifaced applications. Use of ionizing radiations is rapidly growing in many sectors like nuclear reactors, medical diagnostics, nuclear research establishment, agriculture, food irradiation, biological studies, defects detection in metal castings, nuclear medical imaging, space exploration, high energy physics experiments [1], nuclear waste storage sites, aerospace and nuclear industries [2]. There are numerous therapeutic uses of radiations in chemotherapy/radiation therapy for the treatment of tumour, cancer of different organs and other diseases. Therapeutic usage of radiations requires calculated doses for required results for specific time period. Excess exposure to high energy radiation for a longer period is toxic and may lead to serious consequences and multi organ failure in extreme cases. People working in radiation prone areas like nuclear facilities, labs, hospitals and diagnostic centres are under the continuous threat of radiation exposure. This necessitates the usage of radiation shielding materials. Traditional materials used for radiation protection are aluminium (Al), iron, copper (Cu), lead (Pb), bismuth (Bi) and concrete. Lead has been used since inception as radiation shielding material. Though lead has good shielding properties but is categorized as a toxic material [3]. Moreover, it is heavy to carry, has no flexibility and its usage is not environment friendly. Leaded aprons, gloves and other protective garments are heavy to wear and recycling procedure requires a very high cost. Moreover, intensive daily use and lack of careful handling of protective garments may result in structural damages which makes them less effective [3]. These problems can be solved by developing tailored materials using polymers composites [4-9]. There are several reports which confirm the usage of polymer composites as radiation shielding materials [10-13]. Promising features of the polymer are enhanced by the addition of nanofillers. The transition from bulk to nano regime tremendously enhances physical and chemical properties [14-16] of the material which are entirely different from the bulk material. Nano-materials have certain advantages like ease of handling, light weight, non-toxic and high aspect ratio. Nano-composites can be used as radiation attenuating material with suitable choice of fillers. Moreover, loading requirements for the nano-fillers is

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considerable low than the bulk materials. These properties of nano composites must be exploited to protect the environment. Carbon and its allotropes in the nano form have great applications in this regard.

Graphene, a derivative of carbon has been the most fascinating material since its discovery by Geim[17] and has an excellent application potential in various fields. Peculiarity of its structure makes itsutility spectrum quite vast ranging from solar cells, touch less screens, spintronics, super capacitors [18], gas detectors [19-20], superconductors [21-22], drug delivery systems, biomedical applications [23-24], water purification [25], for the removal of radioactive toxins and long-lived manmade radio nuclides from contaminated water [26]. In the recent past carbon, activated carbon, graphite, carbon alloys, fullerenes, single walled carbon nano-tubes (SWCNT), multi walled carbon nano-tubes (MWCNT), nano-diamonds and graphene has been utilized for radioactive waste management [27]. Graphene oxide (GO) has been found to be quite effective in the removal of trans-uranium elements from stimulated nuclear waste solutions [28].Literature mentions that activated carbon and activated carbon impregnated with various metals have been used successfully for water treatment, in chemical industry, food industry and gamma ray shielding[29,30].

Shielding of radiations is closely related to the power of any element to fade the intensity of radiation.MAC, specifies the interaction probability of a radiation beam in matter. According to the International Commission on Radiation Units and Measurements, "It is the quotient dN/N by ρdx where dN/N is the mean fraction of particles that experience interactions in traversing a distance dx in the material of density ρ" [31]. MAC depends upon the density of the material, the photon energy and includes the photo-absorption and scattering effects.The relationship between attenuation and probability of penetration can be expressed by Beer-Lambert law. There are reports of the comparative study of MAC and range determination of different metals [32] using beta source. Mass attenuation coefficient of metals and metals alloys have also been investigated by computational simulations [33]. MAC of transparent barium-bismuth-borosilicate glasses, polymeric blends [34], light weight clay ash [35] building materials [36, 37]for gamma ray energies have been studied using computer simulations. Study of X-ray attenuation coefficients of graphene oxide - PbO - epoxy composites [38] show that better attenuation can be obtained by the introduction of GO in PbO-epoxy composites. There are mentions that GO-Pb_3O_4 hybrid nano-sheets [39]have been used successfully for electromagnetic shielding effects. Nano allotropes of carbon have been investigated as radiation shielding materials [40] and MAC of MWCNT, SWCNT and graphene has been investigated. Polymers and carbon nano-fillers have been reported to be used for radiation protection[41–43]. GOas a filler in metal matrices produces ultra-high-strength joints [44,45] and has high radiation tolerance. GO has a wide absorption bandwidth which makes it favourable for electromagnetic interference shielding[46].

Conventional methods of production of graphene requires harsh chemicals and additives such as dimethyl formamide, hydrazine and sodium borohydride. Keeping the environmental issues and the need of cost-effective radiation shielding materials, an effort has been made to synthesize GSC by a green route [47]. The prepared samples have been characterized by x-ray diffraction, IR spectroscopy, FE-SEM and Raman spectroscopy. Sample discs of different thicknesses have been prepared and radioactive parameters have been obtained from the experimental data.

II. EXPERIMENTAL WORK

2.1 Materials and Methods

Quartz sand was obtained from CLG Export company, Moradabad, UP, India. Graphene oxide was obtained from Ad-Nano Technologies Private Limited, Shimoga, India and having a thickness in nano regime. Concentrated H_2SO_4, and other chemical used in sample preparation are of analytical grade.

2.1.1 Sample preparation

Quartz sand was cleaned by repeatedly washing with water and sonicated for an hours using the probe-sonicator. Measured amount of sonicated sand and sugar (~5%) was mixed and heated on hot plate with magnetic stirrer to form a homogeneous mixture. The mixture was then heated at high temperature (~750 ^{0}C) in electric furnace for about six hours in the presence of nitrogen as explained in reference [47]. The mixture was then treated with concentrated sulfuric acid and rinsed several times until neutral pH water is obtained. Synthesised GSC was dried in vacuum oven at 60 ^{0}C for two hours. Figure 1a. and figure 1b. show the virgin quartz sand and prepared GSC respectively. GSC was compressed by a pallet making machine to form the discs of 1 cm diameter using the pressure pump and a sample holder. Small amount of KBr was added in the samples to retain the shape. Same procedure was followed for making GO discs. Radioactive Sr^{90} disc source obtained from Bhabha Research Atomic Centre (BARC), Mumbai was used in the study. Geiger Muller counter was used to obtain the attenuated intensity emitted by Sr^{90}beta source.

III. RESULTS AND DISCUSSION

XRD studies of prepared GSC shows a well-defined peak at 26.6^0(Figure 2a.). Through XRD studies, the interplanar spacing can be evaluated using Bragg's law. Peak at 2Ө value of 26.6^0suggests a spacing of 3.33 Å and belongs to quartz mineral present in GSC sand [48]. The presence of strong peaks at 2Ө values between 20^0 to 27^0 confirms the synthesis of graphene [49]. Elemental analysis of the GSC has been shown in figure 2b. Carbon has the maximum weight percentage (45.64%) among the constituent elements of GSC. Other major constituents of GSC are O(23.34%), N(17.71), and S(11.82%). Si(0.41%), Cu(0.32%) and Ca(0.6%) are present in lesser concentrations. The greater percentage of carbon in the analysis suggests that quartz sand is appropriately graphitized and encapsulated by sugar.


Fig. 1(a):Virgin quartz sand.


Fig.1(b): Prepared GSC.

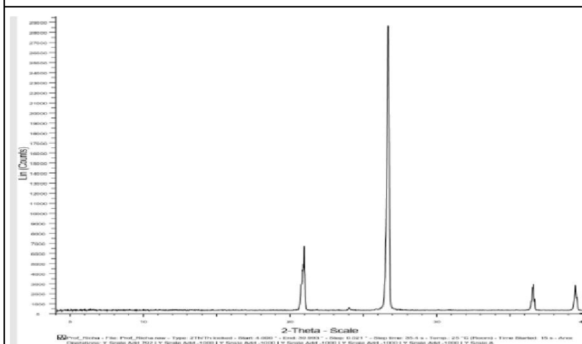

Fig. 2(a): XRD of GSC.

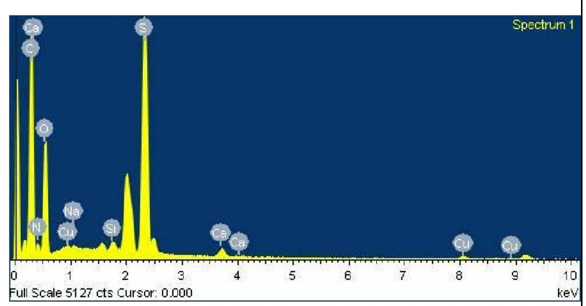

Fig. 2(b): EDAX analysis of GSC.

Raman spectroscopy is a standard non-destructive and excellent tool for the structural characterization of carbon family. The conjugated and double carbon-carbon bonds contribute to high intensities in Raman spectrum of graphene. Laser excitation in graphene causes the Stokes phonon energy shift and gives rise to D (~1350 cm^{-1}) and G (~1580 cm^{-1}) [50] peaks. The position of D peak dependent supon the laser excitation energy [51].G band arises in the first-order scattering from the optical E_{2g} phonons at the Brillouin zone centre resulting from the bond stretching of sp^2 carbon pairs in ring and in chains. D band represents the breathing mode or disorder-induced modes of aromatic rings which arises due to structural defects such as vacancies and grain boundaries. The intensity of the D peak is the measure of the degree of disorder. The intensity ratios of D and G band is less than one (<1) for graphene, and greater than one (>1) for graphene oxide. The details of the G and D bands for the GSC along with the GOhas been shown in the table 1. along with the I_D /I_G ratio. Figure3a. and figure 3b. show the Raman spectra of procured GO and synthesized GSC

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respectively. High intensity of D and G peaks in Raman spectra decides the quality of the sample. Intensity of the D and G peaks are high in the procured GO than that of the synthesised GSC but peak ratio in GO and GSC is approximately the same (~1).

Table 1:Ratio of peak intensities of GO and GSC from Raman Spectroscopy.

Sample Identity	I_D	I_G	I_D / I_G
GO	4882.89	4846.26	1.006
GSC	1693.39	1679.80	1.008

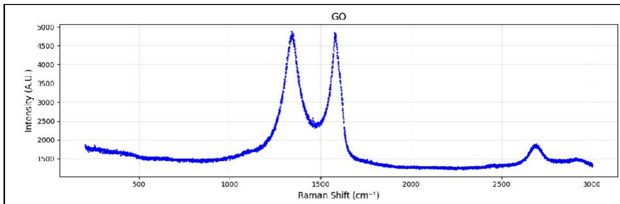

Fig.3(a): Raman spectra of GO

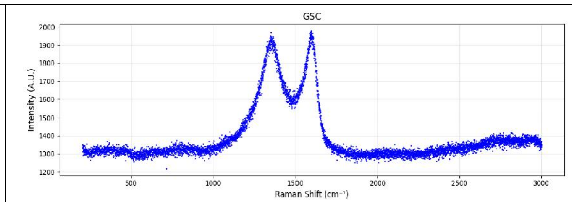

Fig.3(b):Raman spectraof GSC

Infrared (IR) Spectrum of GO and GSC are shown in figure 4a. and figure 4b. respectively. The vertical line on wavenumber axis separates the spectrum it into two regions. The region from zero to 2000 cm^{-1}on x-axis is known as the fingerprint region and gives information about the bonds present in the sample. Thewavenumber beyond 2000 cm^{-1} shows the presence of functional groups in the sample. IR spectra of GO and GSCseems to be quite similar in the functional group region but finger print regions are different. A broad band around 3350 cm^{-1}shows the presence of O-H functional group. This absorbance band in GO seems to be broader than that in GSC. Peak at 470 cm^{-1}in GSC is due to the presence of SiO_2 present in the quartz sand [52]. Peaks at 3540 cm^{-1}, 1650 cm^{-1} and 1575 cm^{-1} in GSC are due to the stretching of O-H, C=C and C-C bonds respectively.The presence of these characteristic peaks confirms the synthesis of GSC [53].

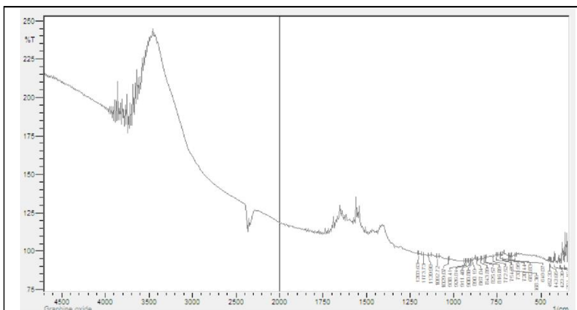

Fig.4(a): FTIR of GO.

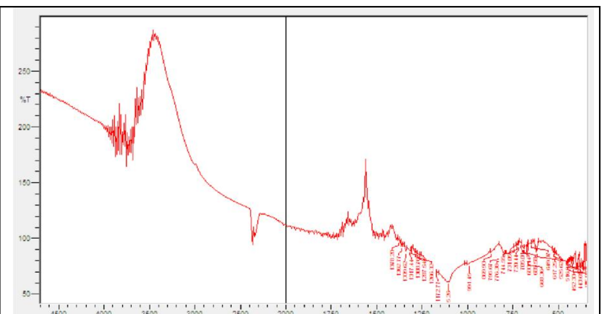

Fig.4(b): FTIR of GSC.

The surface morphology of the samples was studied with a field emission electron microscope with model JEOL JSM-7600F FEGSEM. Figure 5a.and figure 5b. represent the surface morphology of virgin quartz sand and the particles of different sizes are visible. Virgin quartz sand particles have a combination of rough and uniform surface along with the presence of cracks and micropores. After the heat treatment (Figure 5c and figure 5d) the surface morphology of quartz sand changes. The particles seem to be broken and some visible indents are noticed on the surface. This increases the surface area of the particles and provides better opportunity for radiation attenuation. Figure 5e. and Figure 5f. show the morphology of synthesised GSC and agglomerated sheets can be observed through layered structure. Some small sheets and some agglomerated layered large sheets are present in the micrograph.

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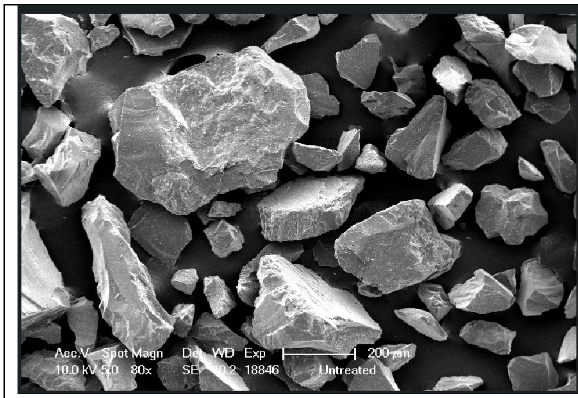

Fig.5(a): SEM of Untreated Quartz sand at lower magnification.

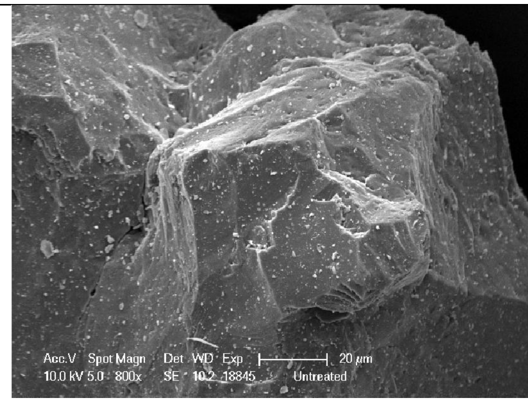

Fig.5(b): SEM of Untreated Quartz sand at higher magnification.

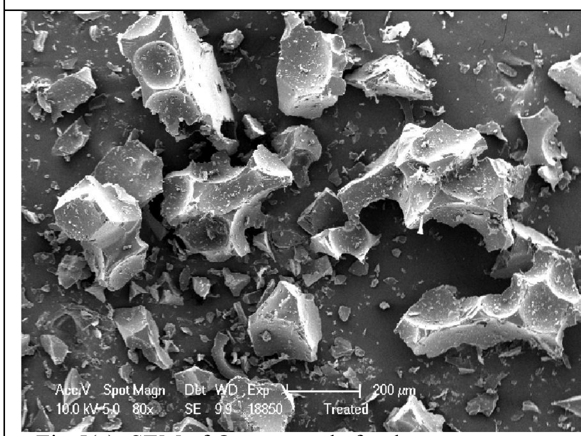

Fig.5(c): SEM of Quartz sand after heat treatment at lower magnification.

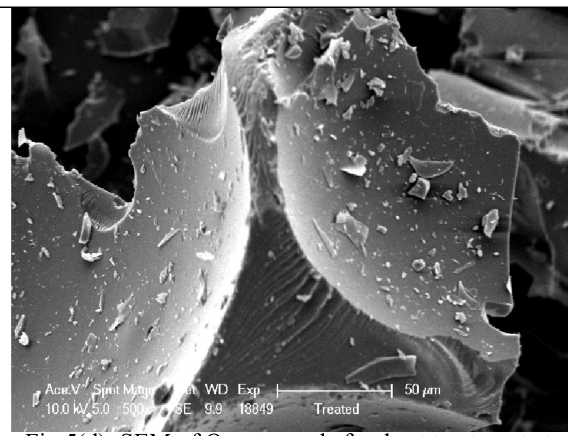

Fig.5(d): SEM of Quartz sand after heat treatment at higher magnification.

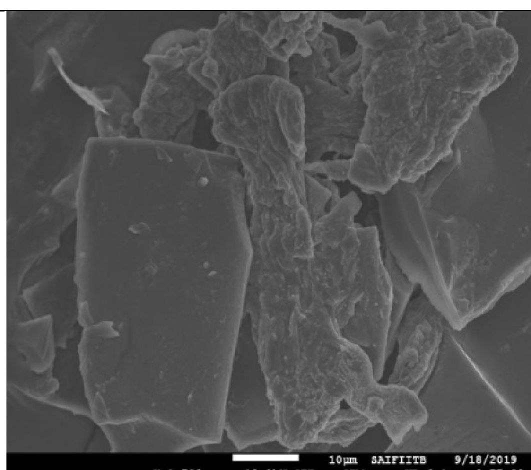

Fig.5(e): SEM of graphitized Quartz sand at lower magnification.

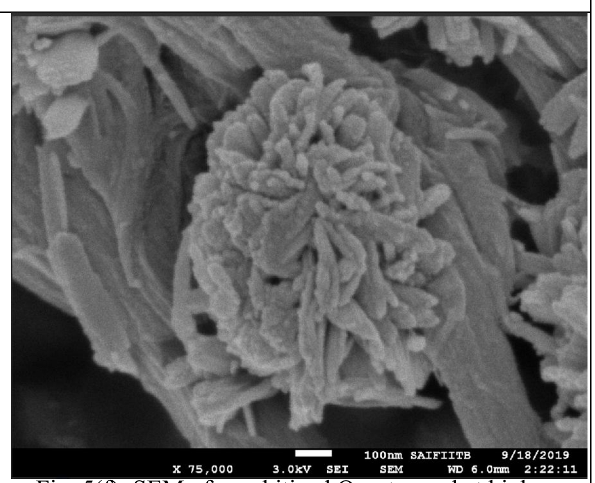

Fig. 5(f): SEM of graphitized Quartz sand at higher magnification.

The attenuated intensity of the prepared and procured samples was measured using a pure beta emitterSr^{90}source, having an energy of 2.27 Mev. For calculation purpose the density of the samples has been taken same as that of the prepared discs. MAC is the measure of the interaction probability of the incident radiation with the unit mass per unit area of the sample.MAC has been evaluated using the Beer-Lambert lawfor GO and GSC using figure 6a. and 6b. respectively. The MAC of GO and synthesized GSC as evaluated from the experimental data comes out to be 1.0696 cm^2/gm and 0.7628 cm^2/gm respectively for 2.27 MeV beta rays. In literature, MAC of several nano-carbon allotropes [39] has been evaluated and found to be 0.497 cm^2/gm and 0.563 cm^2/gm respectively for graphene and multiwalled carbon nano tubes for 0.356 MeV gamma rays. Their study suggests that graphene and MWCNT can be used as a radiation shielding materials. MFP is the average distance travelled by a particle before it suffers collision with the atoms of the matter. In the collisions particle changes direction and may change energy. Half-value layer and ten-value layer is the thicknesses of the material at which the intensity of the incoming radiation reduces to one half (1/2) and one tenth (1/10) respectively of the original intensity. Mean free path,half-value layer and tenth-value layer of GO and GSC for beta particles emitted by Sr^{90} source is listed in table 2 along with LAC, MAC.

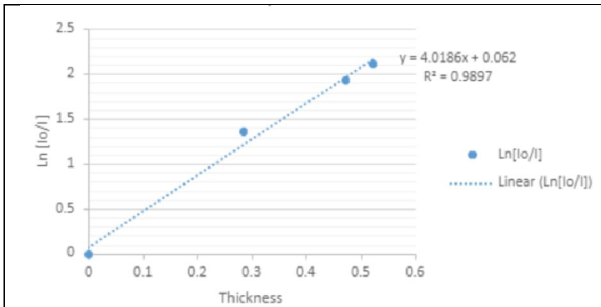

Fig.6(a): Linear attenuation coefficient of GO.

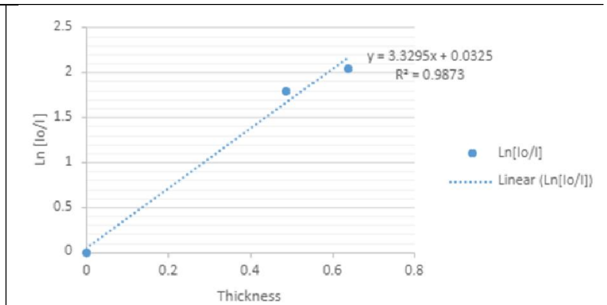

Fig.6(b): Linear attenuation coefficient of GOSC.

Table 2: Radioactive parameters of GO and GSC obtained from the experimental data.

Sample Identity	**μ (Linear attenuation coefficient) (cm^{-1})**	**μ/ρ (Mass attenuation coefficient) (cm^2 /gm.)**	**Mean Free Path (cm.)**	**Half Value Layer (cm.)**	**Tenth Value Layer (cm.)**
GO	4.0186	1.0696	0.2488	0.1725	0.5729
GSC	3.3295	0.7628	0.3004	0.2082	0.6916

IV. CONCLUSION

GSC has been synthesized in a non-hazardous and environmentally safe green manner. X-ray diffraction studies show a well-defined peak at26.6^0 is a representative of quartz sand. IR studies and the presence of bonds confirms the synthesis of GSC. FE-SEM images confirm the layered structure of GSC. Synthesis of GSC is further confirmed by the Raman spectroscopy by the well-defined D and G peaks. MAC has been evaluated for GO and GSC. The value of the MAC suggests that GO and GSC both are having the power to fade the intensity of beta radiations. Which suggests that GO and GSC can be used for can find applications in radioactive waste management, as radiation attenuating/shielding materials and water purification.Moreover, GO and GSC may be used as fillers in protective garment manufacturing in combination of flexible polymers.

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