A multi-energy (2–60 keV) calibration of 200 µm and 400 µm diameter spectroscopic GaAs X-ray photodiodes

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ABSTRACT: Thin (2 µm active layer) spectroscopic p⁺-i-n⁺ GaAs X-ray photodiodes of circular mesa geometry (200 µm and 400 µm diameter; one representative diode of each diameter) have been characterised for their energy response using high-purity X-ray fluorescence calibration samples excited by an X-ray tube, giving energies between 2.1 keV (Au M\(\alpha_1\)) and 21.18 keV (Pd K\(\alpha_1\)), and an \(^{241}\)Am radioisotope \(\gamma\)-ray source (26.3 keV, 59.5 keV). The photodiodes were operated uncooled at +33°C. The 200 µm diameter device’s energy resolution (FWHM) was found to be constant (0.79 keV) and primarily limited by electronics noise at energies between 2.1 keV and 21.18 keV, but it broadened to 0.85 keV at 26.3 keV, and to 1 keV at 59.5 keV. The 400 µm diameter device’s energy resolution (FWHM) was constant (1.1 keV) for photon energies between 4.95 keV and 9.89 keV, but increased to 1.15 keV at 16.62 keV, 1.25 keV at 21.18 keV, 1.3 keV at 26.3 keV and 1.66 keV at 59.5 keV. The broadening of energy resolution (FWHM) observed in both cases is greater than can be attributed solely to increasing Fano noise and is hypothesised to be at least in part due to energy dependent charge trapping. However, for both types of device, the peak charge output from the devices was found to be linearly \((R^2 \geq 0.9999)\) dependent on incident X-ray energy.

KEYWORDS: Solid state detectors; Materials for solid-state detectors; Space instrumentation

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1 Introduction

X-ray photodiodes made from materials with wider band gaps, $E_g$, than silicon ($E_g = 1.12$ eV), such as 4H-SiC ($E_g = 3.26$ eV), Al$_{0.8}$Ga$_{0.2}$As ($E_g = 2.09$ eV) and GaAs ($E_g = 1.43$ eV), offer the ability to operate uncooled at higher temperatures than silicon detectors of the same design due to lower thermally induced leakage currents [1] and better resistance to damage from radiation. While silicon is well established as a material for X-ray photodiodes, alternative compound semiconductor technologies continue to be developed and improved (e.g. SiC [1–11], AlGaAs [12–16] and GaAs [17–20]), with a view to producing radiation-hard spectroscopic X-ray detectors that are able to operate uncooled in high temperature and harsh environments, for use in future space missions and terrestrial applications.

In this paper, we report new results from circular GaAs X-ray photodiodes of two different diameters (200 $\mu$m and 400 $\mu$m) showing their energy resolution across the energy ranges 2.1 keV to 59.5 keV, and 4.95 keV to 59.5 keV respectively, and characterising their charge output variation with incident photon energy. Measurement of the 2.1 keV Au M$\alpha$$_1$ line was not possible with the 400 $\mu$m diode because it is below the low energy noise threshold for this device. The results presented here also represent an extension of the energy range that this type of photodiode has been characterised at: we have previously reported some early results characterising a 200 $\mu$m diameter GaAs photodiode of the same type with an $^{55}$Fe radioisotope X-ray source [20] and $^3$H and $^{14}$C beta particle sources [21]. The results reported here also represent the first report from our laboratory of a 400 $\mu$m diameter GaAs X-ray photodiode.
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Figure 1. An optical microscope image of a set of devices with the same mask design as used in this work. The largest diodes are the 400 $\mu$m diameter devices and the next biggest size devices are the 200 $\mu$m diameter devices.

2 The GaAs photodiodes

GaAs photodiodes photolithographically etched from an MBE-grown wafer (table 1) as part of a joint research programme between University of Leicester and University of Sheffield were mounted in TO-5 packages. The mask design used is shown in figure 1. The p$^+$-side contact metallisation is Au/Zn/Au in the ratios 5 nm/10 nm/200 nm. The metallisation covers 45% and 33% of the active faces of the 200 $\mu$m and 400 $\mu$m diameter photodiodes, respectively. The 200 $\mu$m and 400 $\mu$m diameter devices had capacitances at full depletion (10 V reverse bias) before packaging of 1.94 pF and 7.21 pF, as measured at University of Sheffield using an HP 4275 LCR meter with the AC test voltage signal magnitude and frequency set to 50 mV r.m.s. and 1 MHz. After packaging, they were measured at University of Leicester to have leakage currents of 12.2 pA and 12.9 pA, respectively, at reverse biases of 10 V in a dry N$_2$ environment at a temperature of 21$^\circ$C.

The devices were grown as part of a programme working towards thicker (higher efficiency) devices and monolithic photodiode arrays.

3 Experimental method

A representative diode of each diameter was connected in turn to a low-noise charge-sensitive preamplifier made at University of Leicester. The preamplifier is of a custom design; it has a Vishay Siliconix 2N4416 JFET (capacitance = 2 pF) for the input transistor and does not use a feedback resistor. The diodes were installed within the preamplifier housing, behind a 4 $\mu$m thick Al foil window. To eliminate the possibility of any humidity related effects on the diode, the housing was continually purged with dry N$_2$ throughout the experiments. The preamplifier was connected to an Ortec 571 shaping amplifier (shaping time = 2 $\mu$s) and an Ortec EASYMCA-8k multi-channel analyser (MCA).
Table 1. Layer details of the GaAs wafer from which the diodes were fabricated [20].

| Layer | Material | Thickness (µm) | Dopant | Type | Doping Density (cm$^{-3}$) |
|-------|----------|----------------|--------|------|--------------------------|
| 1     | GaAs     | 0.01           | Be     | p    | 1.0×10$^{19}$            |
| 2     | GaAs     | 0.2            | Be     | p    | 2.0×10$^{18}$            |
| 3     | GaAs     | 2              | Undoped|      |                          |
| 4     | GaAs     | 0.1            | Si     | n    | 2.0×10$^{18}$            |
| 5     | GaAs     | 0.2            | Si     | n    | 2.0×10$^{18}$            |
| Substrate | n$^+$ GaAs |       |        |      |                          |

3.1 X-ray fluorescence measurements

The preamplifier and diodes were installed within a LD Didatic GmbH X-ray apparatus (part number 554 800) with Mo target X-ray tube (part number 554 861) and connected to a goniometer (part number 554 831) to enable precise positioning (figure 2). A custom-made aluminium collimator was used to collimate X-rays from the Mo tube. The goniometer sample stand was positioned at 45° to the collimator. The preamplifier and diodes were positioned at 135° to the collimator, with the diodes centred on the sample stand.

Nine high-purity metal X-ray fluorescence calibration targets of known composition (table 2) were each positioned in turn on the sample stand and a spectrum from each accumulated for 17 hours. The preamplifier and photodiode were powered continuously throughout the accumulation of samples. Each time a new target was installed, the X-ray tube was allowed to stabilise for 3 hours after switching on before accumulation was started. This stabilisation period also enabled each spectrum to be taken at the same temperature: the internal preamplifier temperature stabilises at 33.3°C (as measured by thermocouple thermometer), heated by the proximity of the X-ray tube after this period of time. The rise in internal temperature of the preamplifier from room temperature to stable operating temperature is shown in figure 3. For all the measurements reported here, the X-ray tube voltage and current were 35.0 kV and 1.00 mA, respectively. The process was carried out for both the 200 µm diameter diode and the 400 µm diameter diode.

3.2 Radioisotope γ-ray measurements

For characterisation with the $^{241}$Am radioisotope source (350 MBq, 26.3 keV and 59.5 keV emissions), the source was positioned 3 cm in front of the Mylar window and supported by a custom mount on the sample stand. The same spectrum accumulation procedure was used for $^{241}$Am spectra as was used for the fluorescence sample spectra. The X-ray tube was left on during the $^{241}$Am spectrum accumulation so that the temperature of the photodiode and preamplifier remained +33.3°C.
Table 2. Details of the fluorescence calibration samples used in this work. For clarity, only the emission lines primarily used for calibration are shown for each sample. Emission line energies taken from ref. [22], except Au M$_{\alpha_1}$ which is from ref. [23].

| Material | Purity (%) | Primary line used | Line energy (keV) |
|----------|------------|-------------------|-------------------|
| V        | ≥ 99.8     | K$_{\alpha_1}$    | 4.952             |
| Cr       | ≥ 99.7     | K$_{\alpha_1}$    | 5.415             |
| Mn       | 98.7       | K$_{\alpha_1}$    | 5.899             |
| Cu       | 99.9       | K$_{\alpha_1}$    | 8.048             |
| Zn       | ≥ 99.95    | K$_{\alpha_1}$    | 8.639             |
| Ge       | 99.999     | K$_{\alpha_1}$    | 9.887             |
| Nb       | 99.9       | K$_{\alpha_1}$    | 16.615            |
| Pd       | 99.95      | K$_{\alpha_1}$    | 21.177            |
| Au       | 99.95      | L$_{\alpha_1}$    | 9.713             |
| Au       | 99.95      | M$_{\alpha_1}$    | 2.123             |

4 Results

4.1 Estimate of the relative detection efficiency between 200 $\mu$m and 400 $\mu$m diameter devices

The relative increase in detection efficiency of the 400 $\mu$m diameter diode over the 200 $\mu$m diode was estimated. For each fluorescence calibration sample spectrum obtained with each device, the number of counts at each energy were compared. Where multiple peaks overlapped, the peaks were deconvolved where possible. The effects of deviations from the ideal Gaussian peak shape, for example as would be caused by charge tailing on the low energy side of the peaks, were minimized.
by measuring the number of counts in each peak between the limits of the peak centroid and right hand limit of the peak’s FWHM only. These limits were applied for both sizes of device. Using this method of estimation reduces the need to decipher details of the low energy tailing for each device, assuming any tailing is proportionally the same for each diode size.

The area of the 400 $\mu$m diameter devices is four times that of the 200 $\mu$m diameter devices, however, the metalized area on each does not scale in the same way. The p$^+$-side contact metalization covers 45% of the 200 $\mu$m diameter diode’s face and 33% of the 400 $\mu$m diameter diode’s face. The metallization also attenuates different X-ray energies differently. Because of this, the numbers of counts between the specified limits in each peak were adjusted to take into account the ratio of the unmetallized areas and the transmission through the metallized areas of the X-ray radiation for each energy and calibration sample. The estimated relative efficiencies for each energy are shown in figure 4. The Pd spectra were excluded from the relative efficiency determination because the non-uniform shape of the Pd foil prevented its repeatable precise positioning on the sample stand. $^{241}$Am spectra were excluded because of the small numbers of counts in the 59.5 keV peaks due to the low efficiency of the devices at this energy (quantum efficiency = 0.002, at 59.5 keV).

The predicted relative efficiencies of the 400 $\mu$m diameter diode compared with the 200 $\mu$m diameter diode at each energy are also shown in figure 4.

4.2 Energy resolution of diodes

The energy resolutions of the photodiodes, as measured by the full width at half maximum (FWHM) of the X-ray peaks at 5.9 keV, are 0.79 keV and 1.1 keV, at +33.3°C, for the 200 $\mu$m and 400 $\mu$m diameter diodes, respectively. Mn (K$\alpha$ = 5.9 keV, K$\beta$ = 6.49 keV) fluorescence spectra accumulated using the 200 $\mu$m and 400 $\mu$m diameter diodes are shown in figure 5. At this photon energy, the resolutions achievable are primarily limited by the preamplifier electronics.
Figure 4. The relative efficiency of the 400 $\mu$m diameter (circles) photodiodes compared with the 200 $\mu$m diameter diodes (squares) as estimated using the procedure detailed in the text. The predicted relative efficiencies of the 400 $\mu$m diameter diodes are also shown (red $\times$ symbols).

Figure 5. Mn spectra accumulated using the 200 $\mu$m (solid black line) and 400 $\mu$m (solid grey line) diameter diodes. The dotted lines show the fitted Mn K$\alpha$ (5.89 keV) and K$\beta$ (6.49 keV) X-ray lines (orange dots — 200 $\mu$m diode; red dots — 400 $\mu$m diode). The FWHM at 5.89 keV are 0.79 keV (200 $\mu$m diode) and 1.1 keV (400 $\mu$m diode).

Figure 6 shows the measured FWHM for each diode across the measured energy range. With the 200 $\mu$m diode, the FWHM is broadly constant for photon energies up to 21 keV. With the 400 $\mu$m diode, the FWHM is measured to be constant for photon energies up to 10 keV. At 16.62 keV (Nb K$\alpha_1$) the FWHM of the 400 $\mu$m diode broadened to 1.15 keV, and at 21.18 keV (Pd K$\alpha_1$) it broadened to 1.25 keV. The data point at 26.3 keV (FWHM$_{200\mu m}$ = 0.85 keV, FWHM$_{400\mu m}$ = 1.3 keV) is the 26.3 keV $\gamma$-ray from the $^{241}$Am radioisotope source.

At 59.5 keV ($^{241}$Am) the peak shape observed with the 200 $\mu$m diameter diode deviates from Gaussian with significant low energy tailing distorting the shape on the low energy side, although
the high energy side of the peak can still be relatively well fitted by a 1 keV FWHM Gaussian. Figure 7 shows the $^{241}$Am spectrum obtained with the 200 $\mu$m diameter device, along with an inset detail of the 59.5 keV peak. With the 400 $\mu$m diameter diode, the 59.5 keV ($^{241}$Am) peak has broadened significantly (FWHM = 1.66 keV) but still appears to be quasi-Gaussian (figure 8). In both cases, because of the low efficiency of the diodes (quantum efficiency = 0.002, at 59.5 keV), there are few counts in the peaks despite the 17 hour accumulation time. The small numbers of counts in each $^{241}$Am spectrum makes conclusive analysis difficult; it is conceivable that with more counts, the spectrum obtained with the 200 $\mu$m diameter diode would also show a more Gaussian and better defined peak, analogous to that seen with the 400 $\mu$m diameter diode.

If the system were Fano limited, the FWHM would be expected to broaden with increasing photon energy, $E$, as per the well-known equation (4.1)

$$\Delta E [\text{eV}] = 2.355 \omega \sqrt{\frac{FE}{\omega}},$$

(4.1)

where $\Delta E$ is the FWHM, $\omega$ is the electron-hole pair creation energy and $F$ is the Fano factor.

The Fano limited energy resolution (FWHM) of a GaAs photodiode is predicted to be $\approx 128$ eV at 5.89 keV, $\approx 243$ eV at 21.177 keV and $\approx 407$ eV at 59.5 keV, assuming $\omega = 4.184$ eV and $F = 0.12$ [24]. At 21.18 keV (Pd K\alpha$_1$), taking into account the electronics noise, experimental FWHM of 0.82 keV and 1.12 keV would be predicted for the 200 $\mu$m and 400 $\mu$m diameter diodes, respectively, where the electronic noise contribution has been determined by assuming that at 5.9 keV all spectrum broadening beyond the Fano limited case is due to the electronics noise (i.e. by assuming that there is no significant charge trapping at 5.9 keV, and hence that the electronic noise equivalent charge is equal to 79 e$^-$ r.m.s. and 111 e$^-$ r.m.s. for the 200 $\mu$m and 400 $\mu$m
Figure 7. $^{241}$Am spectrum accumulated over 17 hours with the 200 $\mu$m diameter diode. The data points have been smoothed (mean average over the 4 lower and higher energy nearest neighbour channels). Inset: the 59.5 keV peak fitted with a 1 keV FWHM Gaussian centred on 59.5 keV (blue dashes).

Figure 8. $^{241}$Am spectrum accumulated over 17 hours with the 400 $\mu$m diameter diode. The data points have been smoothed (mean average over the 4 lower and higher energy nearest neighbour channels). Inset: the 59.5 keV peak fitted with a 1.66 keV FWHM Gaussian centred on 59.5 keV (blue dashes).

diodes, respectively). At low energies, the broadening of the peaks with increasing energy, due to increasing Fano noise, cannot be seen in the experimental data because of the dominance of the electronics noise.

The broadening which is experimentally observed at higher energies is too great to be explained solely by increasing Fano noise. For example, the broadening seen in the diodes’ peaks at 59.5 keV in the $^{241}$Am spectra ($\text{FWHM}_{200\mu m} = 1$ keV, $\text{FWHM}_{400\mu m} = 1.66$ keV) is much larger than that which can be attributed to this mechanism, which predicts FWHM including electronics noise of 0.88 keV and 1.17 keV with the 200 $\mu$m and 400 $\mu$m diodes, respectively, where the electronics noise contribution has been determined by assuming that at 5.9 keV all spectrum broad-
The measured FWHM and the predicted FWHM (assuming that all spectrum broadening beyond the Fano limited case is due to electronics noise rather than charge trapping noise) for the 200 µm and 400 µm diodes. The Au Lα1 peak \((E = 9.71\,\text{keV})\) is not included due to the difficulty of fully deconvolving the many peaks in this region. The Au Mα1 peak \((E = 2.1\,\text{keV})\) is not included for the 400 µm diode because it is below the low energy noise threshold for this device.

| Material | Line energy (keV) | 200 µm diode measured FWHM (keV) | 200 µm diode predicted FWHM (keV) | 400 µm diode measured FWHM (keV) | 400 µm predicted FWHM (keV) |
|----------|------------------|----------------------------------|----------------------------------|----------------------------------|-----------------------------|
| Au       | 2.123            | 0.79                             | 0.78                             | -                                | -                           |
| V        | 4.952            | 0.79                             | 0.79                             | 1.10                             | 1.10                        |
| Cr       | 5.415            | 0.75                             | 0.79                             | 1.10                             | 1.10                        |
| Mn       | 5.899            | 0.79                             | 0.79                             | 1.10                             | 1.10                        |
| Cu       | 8.048            | 0.79                             | 0.80                             | 1.10                             | 1.10                        |
| Zn       | 8.639            | 0.79                             | 0.80                             | 1.10                             | 1.10                        |
| Ge       | 9.887            | 0.79                             | 0.80                             | 1.10                             | 1.11                        |
| Nb       | 16.615           | 0.79                             | 0.81                             | 1.15                             | 1.11                        |
| Pd       | 21.177           | 0.79                             | 0.82                             | 1.25                             | 1.12                        |
| ²⁴¹Am    | 26.3             | 0.84                             | 0.83                             | 1.30                             | 1.13                        |
| ²⁴¹Am    | 59.5             | 1.00                             | 0.88                             | 1.66                             | 1.17                        |

The broadening of the peaks beyond the predicted values, and the charge tailing on the low energy side of each 59.5 keV peak may be attributable to charge trapping, but other experiments, including obtaining spectra at this energy with more counts, to improve the statistics, would be required to verify whether this is the cause.

### 4.3 Energy response linearity

To determine the diodes’ charge output responses as functions of photon input energy, the positions of the X-ray peaks from the fluorescence calibration targets and the 59.5 keV \(\gamma\)-ray peak from the \(^{241}\text{Am}\) radioisotope source (in terms of MCA channel numbers) were plotted against the accepted energy values for those peaks. The results can be seen in figure 9.

The charge outputs of the 200 µm and 400 µm diameter diodes as functions of input photon energy are both highly linear \((R^2\text{ values of } 0.9999\text{ and } 1.0000\), respectively\) and can be represented by the equations

\[
y_{200\mu m} = 126.39E - 1.1574
\]
Figure 9. The charge output linearity response of the 200 µm (squares) and 400 µm (circles) diameter diodes as a function of energy over the range 2–60 keV.

\[ y = 126.39E - 1.1574 \]
\[ R^2 = 0.9999 \]

\[ y = 86.118E + 10.701 \]
\[ R^2 = 1 \]

where \( y \) is the centroid MCA channel number of the X-ray peak of energy \( E \), in keV.

The two different sizes of diode have different response functions because the change in capacitance of the device changes the response of the pre-amplifier (larger capacitance detectors reduce the preamplifier gain).

5 Discussions, conclusions and further work

Circular mesa GaAs p\textsuperscript{+}-i-n\textsuperscript{+} photodiodes of two diameters (200 µm and 400 µm) for photon counting X-ray spectroscopy have been characterised over the energy ranges 2.1 keV to 59.5 keV, and 4.95 keV to 59.5 keV, respectively.

The relative efficiency improvement due to the larger area of the 400 µm photodiode compared with the 200 µm diameter diode was experimentally estimated from X-ray spectra collected with the two devices. The results returned from the method of estimation were consistent (within their error bars) with those predicted from the geometrical areas of the devices and the sizes of the metal contacts covering their faces. At lower energies (< 8.6 keV) the agreement between estimated relative efficiency and predicted relative efficiency was good, but at higher energies the agreement diverged. This was due to the difficulty of fully deconvolving the peaks in those spectra, in order to determine the number of counts with in them.

At 5.9 keV, the FWHM of the 200 µm and 400 µm diameter photodiodes were measured to be 0.79 keV and 1.1 keV, respectively, both at a temperature of +33.3°C. At 59.5 keV, the FWHM of both devices increased to 1 keV and 1.66 keV, respectively. The increases in these FWHM are greater than that which can be explained through the predicted increase in Fano noise with increasing energy alone. Furthermore, there is significant low energy charge tailing on the 59.5 keV peaks,
which is particularly prominent in the $^{241}$Am spectrum obtained with the 200 $\mu$m device. The increase in FWHM with photon energy and charge tailing is suggestive of charge trapping processes, which can be particularly significant at higher energies [25]. A characterisation using a greater number of discrete energy lines, particularly in the region 30 keV to 60 keV (and beyond), would permit the dependence of the FWHM on incident photon energy to be more closely investigated with a view to determining whether the broadening is consistent with the form for noise due to trapping, $r$, which has been previously observed by other researchers as:

$$ r = a_1 E^{\alpha_2}, $$ (5.1)

where $E$ is the incident photon energy and $\alpha_1$ and $\alpha_2$ are semi-empirical constants derived by best fitting [25].

The shapes of the 59.5 keV peaks are also likely a consequence of incomplete charge collection and possibly a consequence of the disparity between electron and hole mobilities in GaAs [26, 27]. The electric field profile within photodiodes is also known to modify peak shapes [26], and the transient effects on the field profile of creating (on average) $> 14,000$ electron-hole pairs at the absorption of each 59.5 keV photon may also have an influence on the local field. Another potential factor is charge from photons absorbed in the 350 $\mu$m thick $n^+$ substrate, diffusing back to the active region of the detector. However, the hole mobility in GaAs is known to be low ($400 \text{ cm}^2 \text{V}^{-1} \text{s}^{-1}$) [25] so the fraction of charge reaching the active region from the substrate may be relatively small.

A detailed analysis of these effects would be interesting, and we hope to report on it in future. It would also be highly beneficial to obtain $^{241}$Am spectra with longer accumulation times (or a more active source) in order to increase the number of counts in the 59.5 keV peaks and improve the peaks’ statistics.

Linearity of response with varying incident energy is important for spectroscopy since precise determination of the energy of the detected photon is necessary in order for the characteristic X-ray emission line which gave rise to that photon to be identified. To investigate this aspect of the devices, the position of the main peaks observed in the spectra (in terms of MCA channel number) were plotted against the accepted energy values for those peaks (figure 9). The result showed linear ($R^2 = 0.9999$ and $R^2 = 1.0000$) responses for both the 200 $\mu$m and 400 $\mu$m diameter devices, respectively, over the energy ranges 2.1–59.5 keV and 4.95–59.5 keV, respectively.

In the absence of the data showing the greater than expected change in FWHM with incident photon energy, the apparent linear nature of the diodes’ responses may have been concluded to be indicative of a high degree of charge collection efficiency from the devices. This could have been said to be due to the thinness of the detectors, the high material quality from which the didoes were fabricated, or a combination of the two, since significant charge trapping would have been expected to have manifested itself by showing deviation from linear form in the charge output response. However, in light of the FWHM variation with photon energy, the true picture is more complicated.

The results presented here show the first multi-energy characterisation of these GaAs X-ray photodiodes. Despite the intricacies of the detectors’ responses at energies $> 10$ keV, the diodes are remarkably consistent at softer energies indicating their suitability for soft X-ray spectroscopy at modest (0.79 keV) energy resolution (electronics limited), and further suggesting that development of GaAs is promising for future room temperature (and hotter) X-ray detectors.
In future publications, we plan more in depth analysis of the devices presented here, as well as similar characterisations of thicker devices developed as part of our on-going programme. Such thicker detectors will increase the device quantum efficiency, and this future work together with the results here, and the previously presented results and other progress (such as the production of detector arrays) continue to improve the technology readiness level of these types of devices for future space and terrestrial applications.

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