Excellent work!

Monday, October 14, 2024

Good! Also keep in mind that if the signal-to-noise ratio is too low, the doublets might be difficult to see

Jigsaw 1B

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Problem 1

In the absence of a broadband decoupling sequence, the coupling between ^{1}H and ^{13}C can be seen.

^{13}C has a natural abundance of 1.1%. Therefore, 1.1 % of ^{1}H will couple with ^{13}C, while the others ^{1}H are in an environment with ^{12}C which is not active (because its nuclear spin is 0).

Therefore, 1.1% of ^{1}H will give rise to a doublet and the rest will be a singlet.

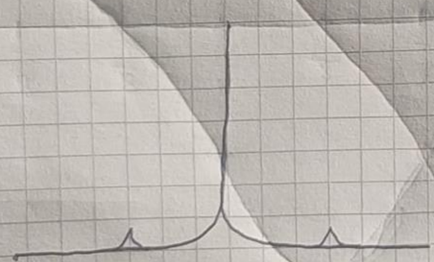

Figure 1. *Problem 1*

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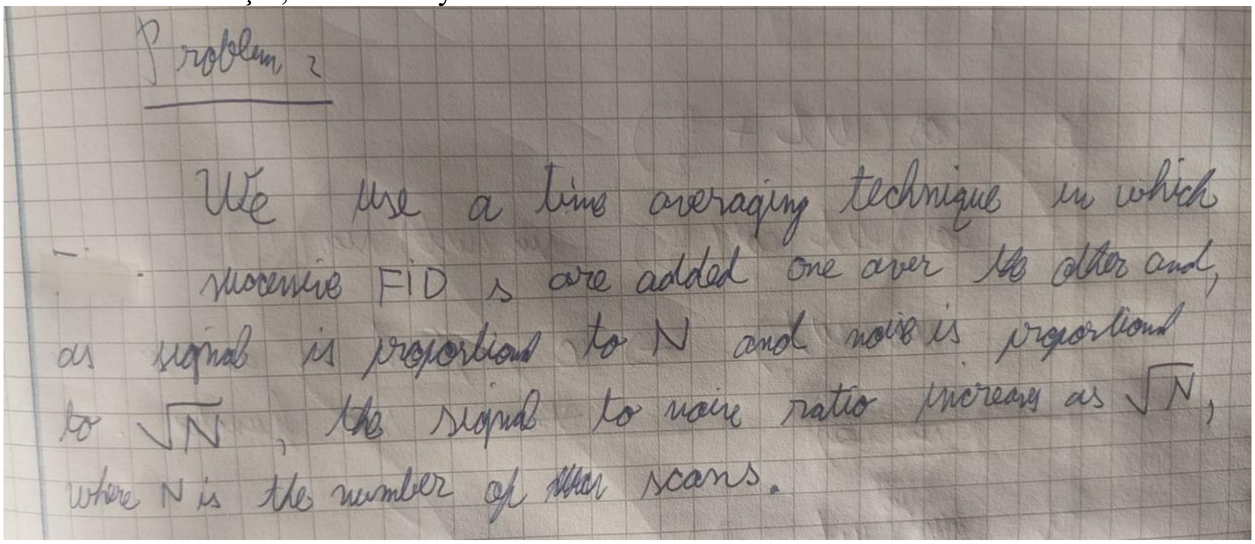
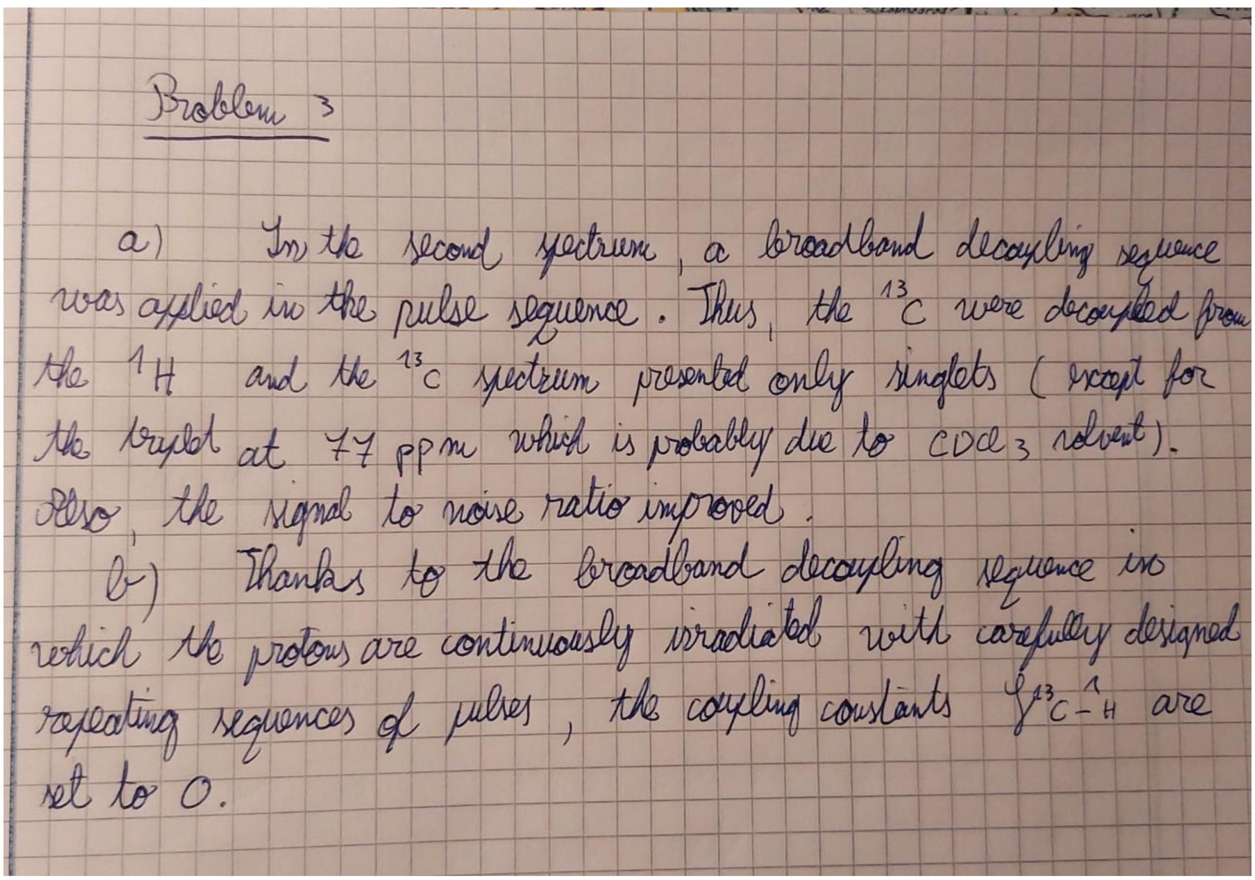


Problem 2

We use a time averaging technique in which successive FID s are added one over the other and, as signal is proportional to N and noise is proportional to $\sqrt{N}$, the signal to noise ratio increases as $\sqrt{N}$, where N is the number of scans.

Figure 2. *Problem 2*



Problem 3

a) In the second spectrum, a broadband decoupling sequence was applied in the pulse sequence. Thus, the ^{13}C were decoupled from the ^{1}H and the ^{13}C spectrum presented only singlets (except for the triplet at 77 ppm which is probably due to $CDCl_3$ solvent). Also, the signal to noise ratio improved.

b) Thanks to the broadband decoupling sequence in which the protons are continuously irradiated with carefully designed repeating sequences of pulses, the coupling constants $J_{^{13}C-^{1}H}$ are set to 0.

Figure 3. *Problem 3.*

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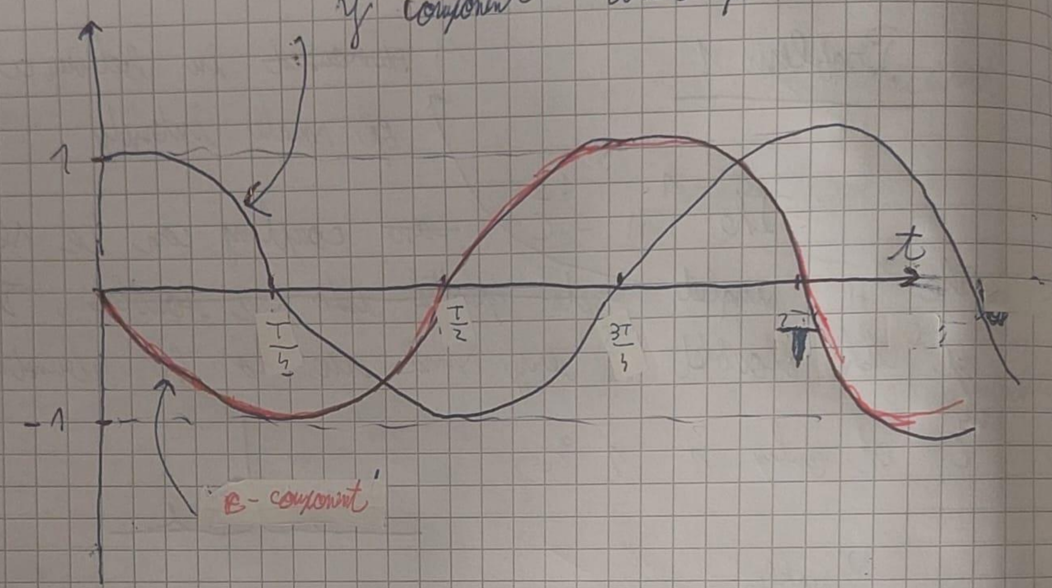
4. T is the period; the plot is done

a. for a vector rotating in a circle of radius 1



$$x = \cos\left(\omega t + \frac{\pi}{2}\right) =$$

$$= \cos(\omega t)\cos\left(\frac{\pi}{2}\right) - \sin(\omega t)\sin\left(\frac{\pi}{2}\right) =$$

$$= -\sin(\omega t)$$ $\Rightarrow$ x component is a ~~mean~~ minus ~~sinus~~ sine function

$$y = \sin\left(\omega t + \frac{\pi}{2}\right) =$$

$$= \cos(\omega t)\sin\left(\frac{\pi}{2}\right) + \sin(\omega t)\cos\left(\frac{\pi}{2}\right) =$$

$$= \cos(\omega t)$$ $\Rightarrow$ y component is a cosine function

Figure 4. *Problem 4 a.*

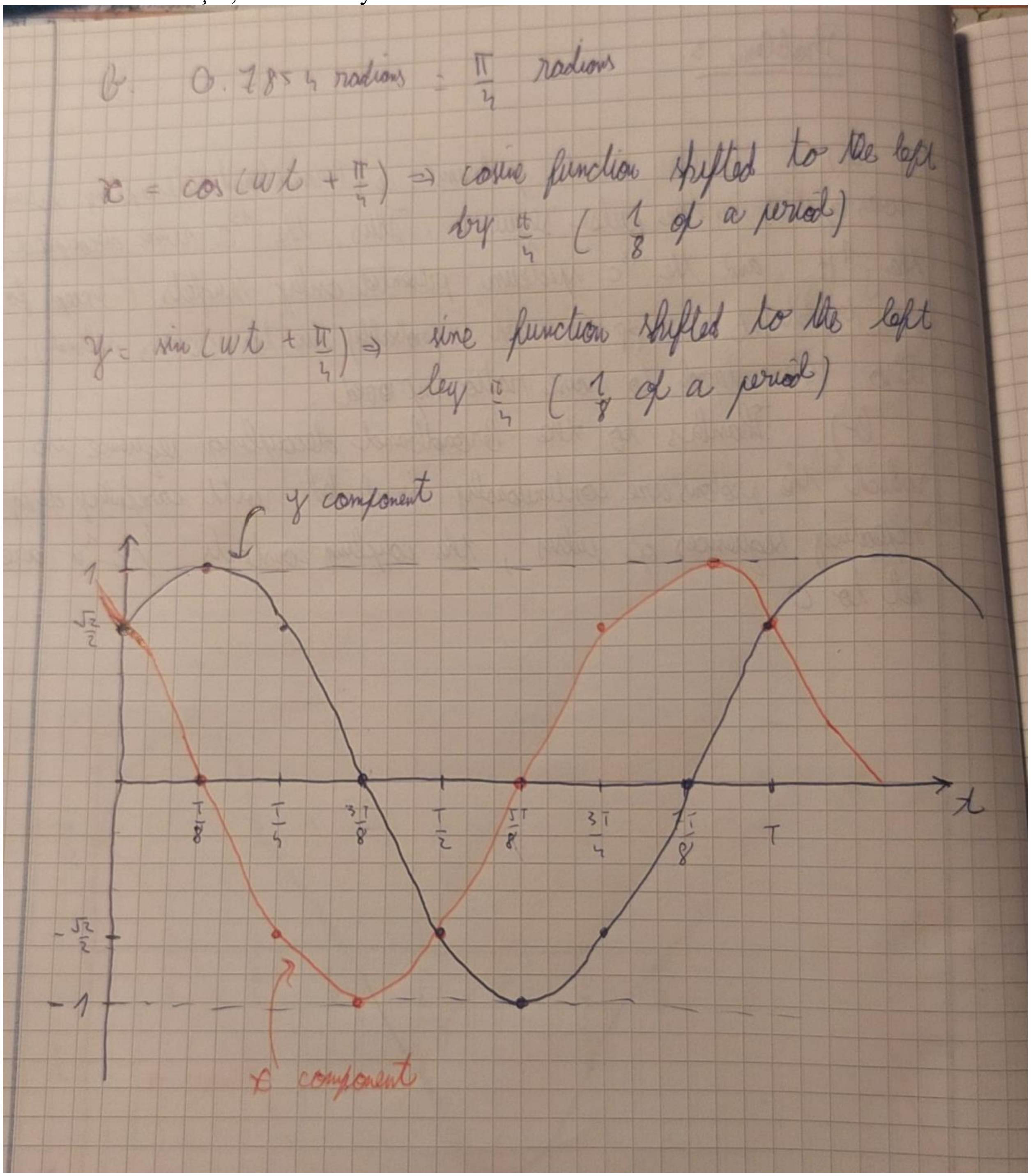


Figure 5. *Problem 4 b.*

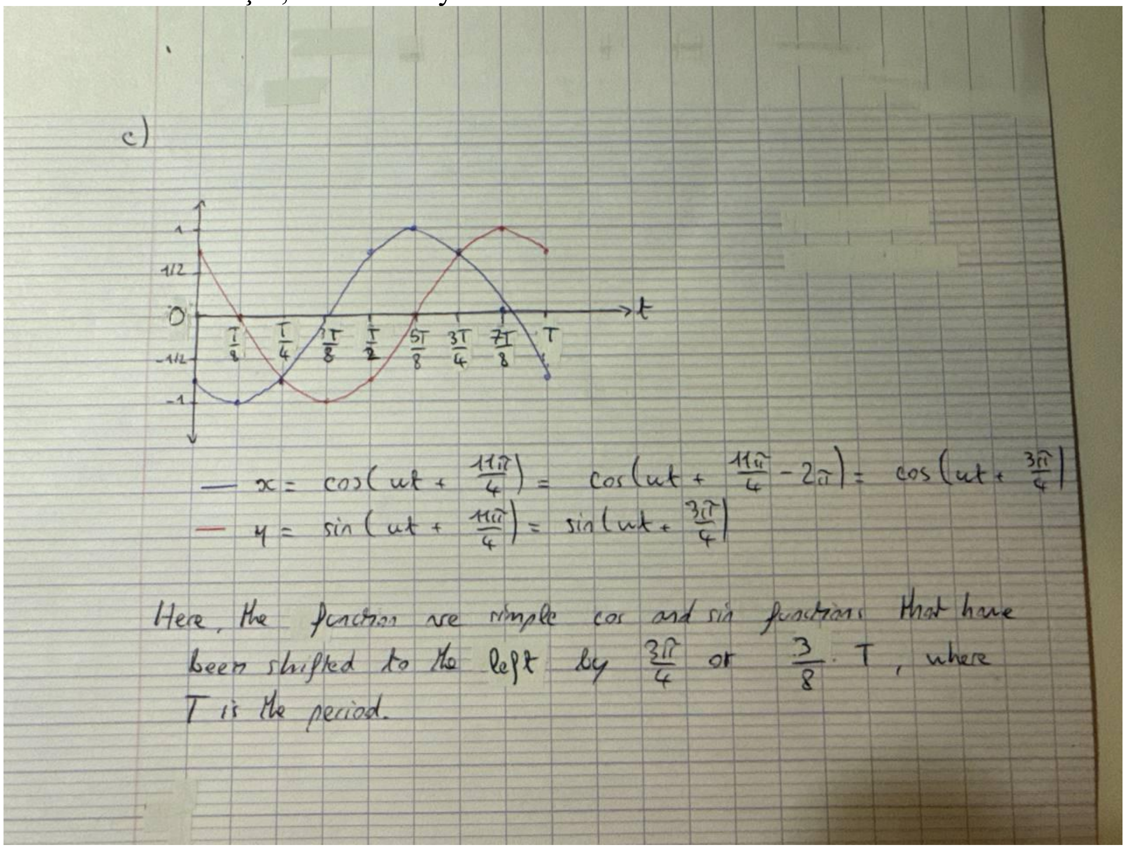


Figure 6. *Problem 4 c.*

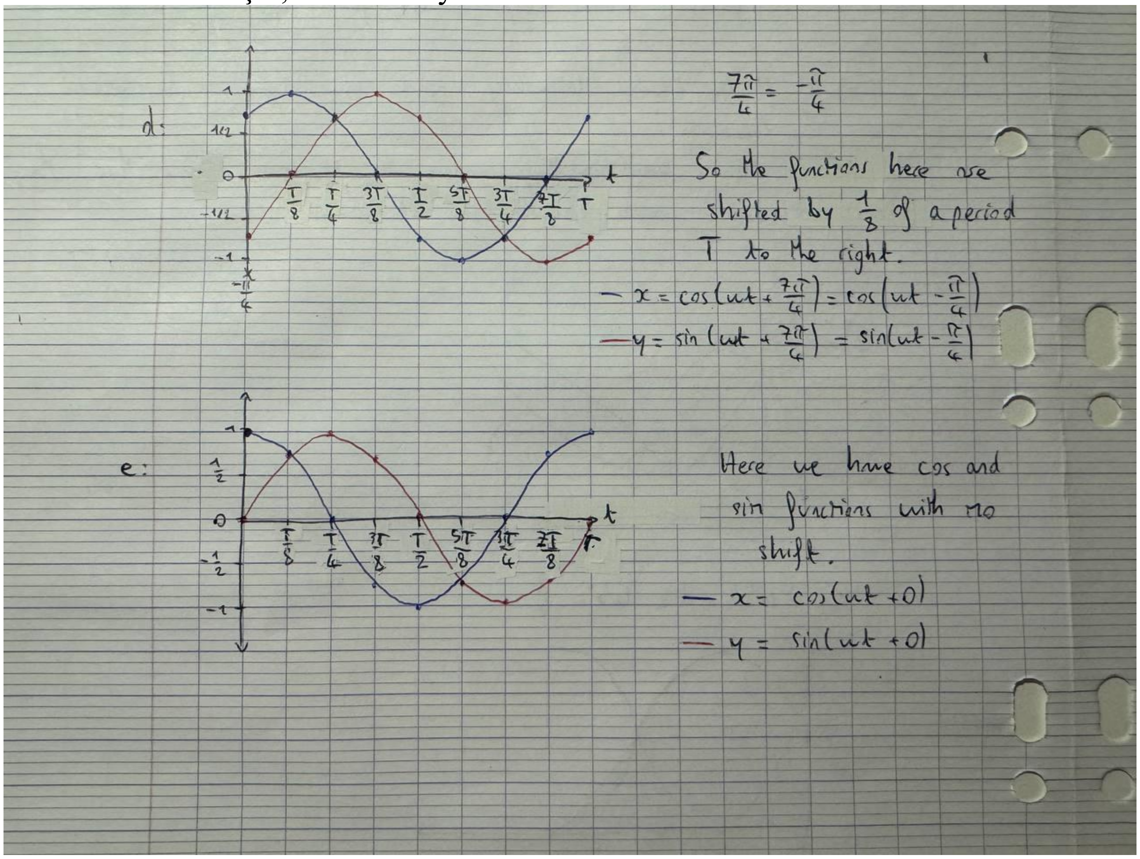


Figure 7. *Problem 4 d+e.*

Good job!

JIGSAW 1B

October 14 2024

• Question 1

Good! Also keep in mind that if the signal-to-noise ratio is too low, the doublets might be difficult to see

We know that *'Scalar coupling can occur between any magnetic nuclei which are reasonably close on the bonding network'* [chapter 2.4.1 Heteronuclear NMR and Broadband decoupling, *Understanding NMR spectroscopy, second edition*, James Keeler]. The natural abundance of the carbon 13 atom is 1.11 % and it has a nuclear spin of $\frac{1}{2}$ like hydrogen, making it NMR-active. We can therefore observe heteronuclear ^{1}H-^{13}C coupling in a $CHCl_3$ sample at natural abundance. This coupling would appear as very small signal next to then main uncoupled signal.

• Question 2

To improve the signal to noise ratio, we use *time averaging*, that is repeating several time the same measurement, then adding together the *free induction decay signal* (the received response of the sample). The signal add linearly, while the noise average itself and grows by the square-root of the number of measurements.
Over N measurements, the signal to noise ratio grows by $\frac{N}{\sqrt{N}} = \sqrt{N}$.

• Question 3

a) When recording ^{13}C NMR spectra, scalar couplings between ^{1}H-^{13}C and ^{13}C-^{13}C nuclei can often be observed, resulting in complex, broad multiplet patterns.

In the case of the two quinine spectra:

- The first spectrum displays several multiplets, indicating the presence of both ^{1}H-^{13}C and ^{13}C-^{13}C couplings.
- The second spectrum shows only singlets, suggesting it was recorded with ^{1}H-^{13}C couplings removed.

To suppress heteronuclear couplings during ^{13}C acquisition, the pulse sequence was modified to include a *broadband proton decoupling* sequence.

b) This change eliminates the multiplets because this broadband proton decoupling sequence involves a designed continuous irradiation that selectively targets ^{1}H nuclei, effectively reducing $J_{(^{13}C-^{1}H)}$ to zero, thereby simplifying the spectral analysis. Indeed, only single peaks will be visible on the spectra at each shift.

Missing the second part of the question (comment on whether they are simple sine/cosine functions) - see above solutions

- ## Question 4

For the following plots : $r\sin(\omega t + \beta)$ and $r\cos(\omega t + \beta)$ were plot choosing arbitrarily $r = 1$ and $\omega = 1.5\,\text{rad/s}$.

x and y are swapped for a and b

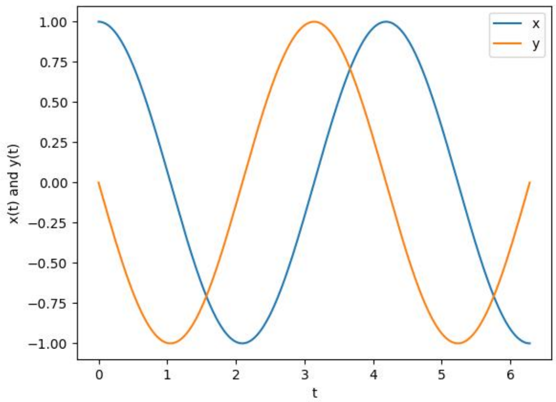


Figure 1: Plot a : $\phi = 90°$

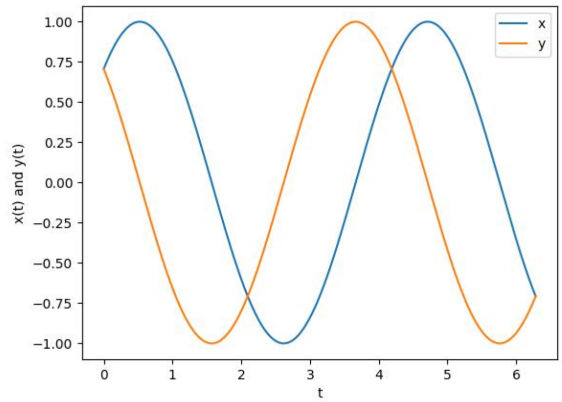


Figure 2: Plot b : $\phi = 0.7854$ rad

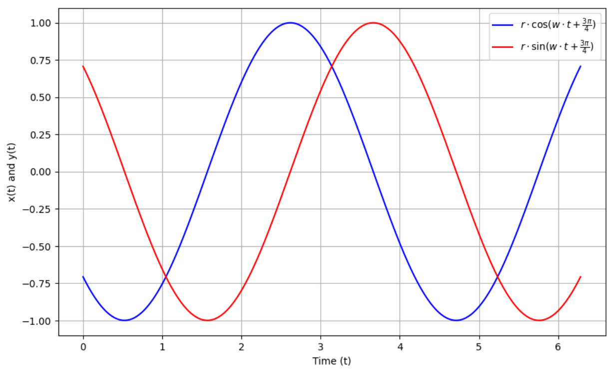


Figure 3: Plot c : $\phi = 495°$

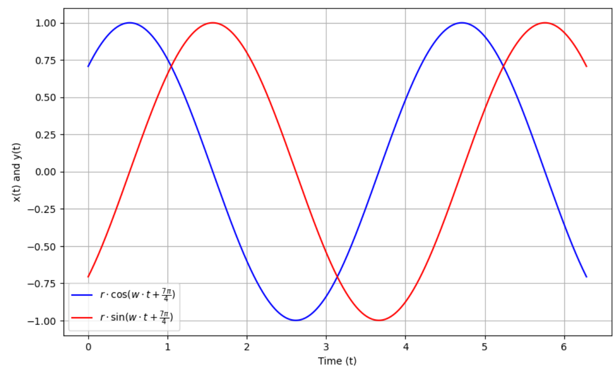


Figure 4: Plot d : $\phi = 7\pi/4$ rad

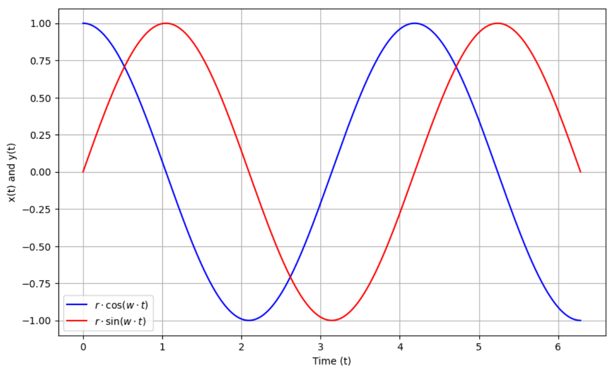


Figure 5: Plot e : $\phi = 0°$

Which is the x component and y component for these 3?