

Sub-surface H₂S detection by a Surface Acoustic Wave passive wireless sensor interrogated with a ground penetrating radar

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Synthesis of complexes

53 mmol of γ -butyrolactone (or ϵ -caprolactone) and 53 mmol of KOH are dissolved in 150ml of ethanol and 30 ml of H₂O. The mixture is heated at reflux for 1h. Then, 26 mmol of Pb(NO₃)₂ in 40 ml of H₂O are added. Then, the mixture is heated at reflux for 20 min. The solvents were removed under reduced pressure. The crude solid is washed in 50 ml of hot ethanol, to give the expected complexes.

Lead(II) 4-hydroxy-butanoate

¹H NMR (300 MHz, D₂O) δ = 3.50 (t, J=7.4Hz, 4H), 2.14 (t, J=7.4Hz, 4H), 1.70 (q, J=7.4HZ, 4H). ¹³C NMR (75 MHz, D₂O) δ = 183.1 (C=O), 61.6 (C-OH), 34.1, 28.4.

Lead(II) 6-hydroxy-hexanoate

¹H NMR (300 MHz, D₂O) δ = 3.57 (t, J=6.5Hz, 4H), 2.19 (t, J=7.40Hz, 4H), 1.66-1.55 (m, 8H), 1.66-1.55-1.50 (m, 4H). ¹³C NMR (75 MHz, D₂O) δ = 184.1 (C=O), 61.5 (C-OH), 38.3, 31.1, 25.0, 24.9.

SAW device fabrication

Ground Penetrating RADAR operating frequency is a tradeoff between antenna dimensions and interrogation range: reaching the 1-3 m depth targeted to reach the water table or the sub-surface infrastructure requires operating in the 100 to 400 MHz range. Thus, YXl/128° lithium niobate acoustic delay lines were manufactured. With a wavelength of 40 μ m and a metal thickness of 2.5% wavelength, the L = 2 to 3 mm long acoustic path introduces a two way trip phase rotation of $2 \times L \times 360 \times f / c = 36180$ to 54270° phase rotation or a differential phase accumulation of 18090° considering the Rayleigh wave velocity $c = 3980$ m/s and an operating frequency of $f = 100$ MHz, or a differential phase rotation of 72360° for the same geometry of a sensor operating at 400 MHz. In order to avoid damping the Rayleigh wave, the organic sensing layer will be designed as thin with respect to the wavelength, aimed at varying the acoustic velocity without excessive acoustic losses: a thickness of about 1% wavelength is considered, yielding 100 to 500 nm thick adlayers for devices operating in the 100 to 400 MHz range.

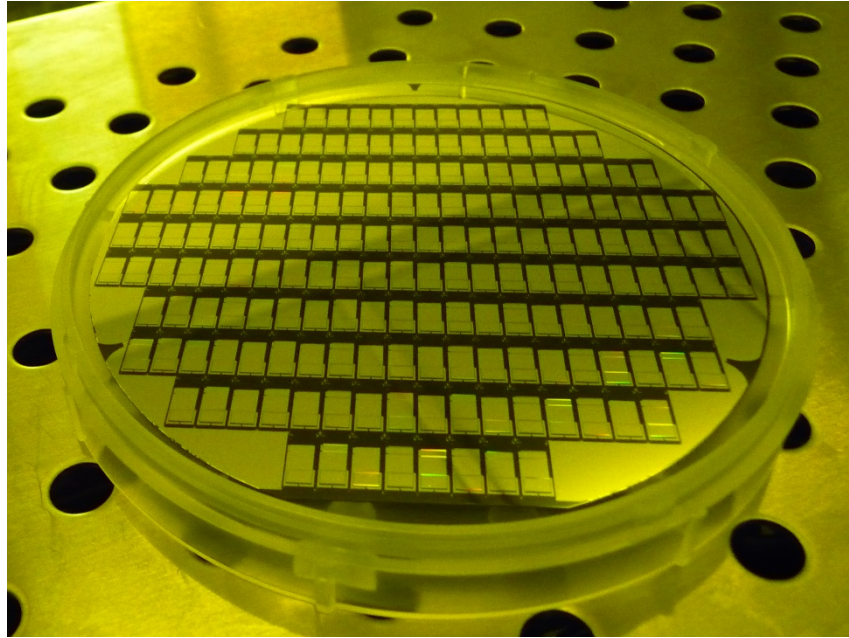


Figure S1. A 4" wafer composed of 200 SAW components completely covers by a solid layer (thickness: 250nm) of the tailored-resist containing the Pb(II)-site.



Figure S2. The sand-pit for the demonstration of sub-surface wireless sensing (left). The GPR is over the sand-pit and the sensor is located in a pipe where H₂S is injected into the pipe as gas phase (right) at different depths (from 0.2 to 1m).

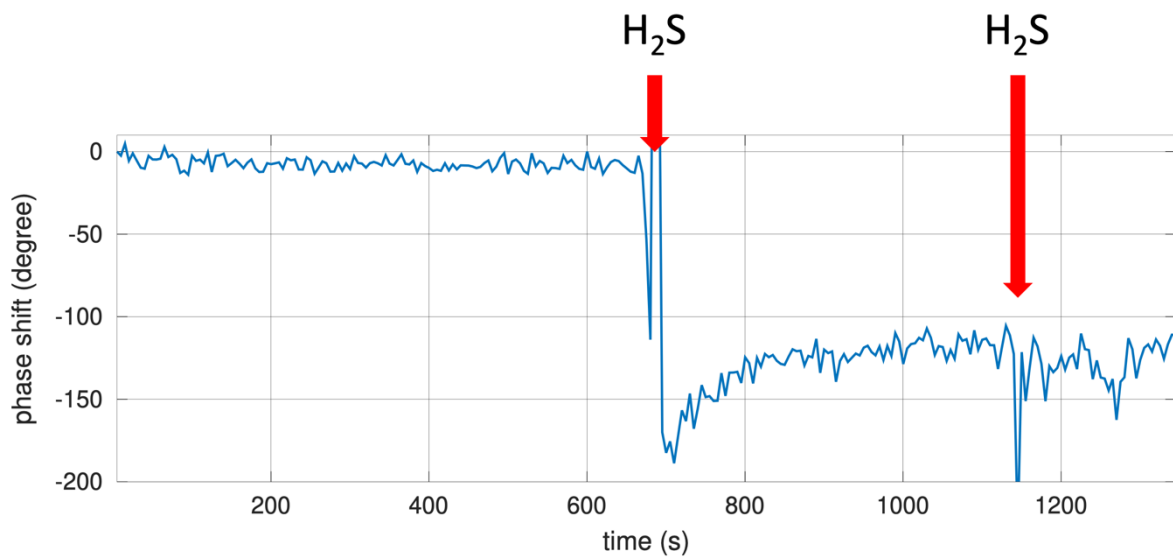


Figure S3. 200-MHz sensor located 0.3-m deep in sand: H₂S is generated at times 695 s and 1150 s. The lack of irreversible phase variation upon the second exposure indicates that the reactive sites were saturated upon the initial exposure.

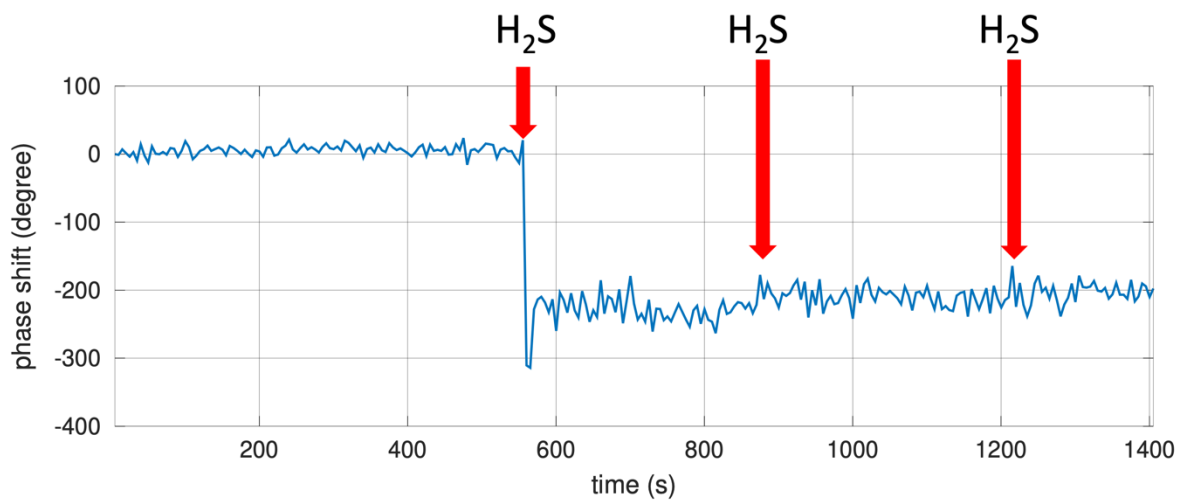


Figure S4. 200-MHz sensor located 0.4-m deep in sand: H₂S is generated at times 565 s, 875 and 1235 s. The lack of irreversible phase variation upon the second and third exposures indicates that the reactive sites were saturated upon the initial exposure.