Electronic supplementary materials *Mendeleev Commun.*, 2022, **32**, 377–378

Molecular structure and photocatalytic properties of the pentaphenylantimony–3,5-dinitrosalicylic acid reaction product

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The IR spectra were recorded on an IR-Fourier spectrometer Shimadzu IR Affinity-1S using the KBr pellet. The ^{1}H NMR (400 MHz) and ^{13}C (126 MHz) spectra were recorded on Bruker DRX-400 and BrukerAvance II spectrometers. Tetramethylsilane was used as the standard. X-ray diffraction study of suitable single crystal **1**·PhMe was accomplished on a Bruker D8 QUEST diffractometer (Mo-K_α radiation, λ 0.71073Å, graphite monochromator). The UV-visible spectrum of diffuse reflectance has been obtained in the range 200−800 nm by a Shimadzu UV2700 UV spectrometer. The suspension was radiated by an Osram high pressure mercury lamp with 125 W power.

*Tetraphenylstibonium 3,5-dinitrosalicylato-O,O'-tetraphenylstibotate, toluene solvate, **1**·PhMe.* A mixure of pentaphenylantimony (0.25 g, 0.5 mmol) and 3,5-dinitrosalicylic acid (0.057 g, 0.25 mmol) in toluene (3 ml) was placed in a glass ampoule and sealed. The mixture was heated in a water bath for 1 h. The solvent was removed to leave 0.24 g (83%) of yellow crystals, m.p. 199 °C (decomp.). IR-spectrum (ν, см$^{-1}$): 1624 (C=O), 1325(C−O), 540(Sb−C), 444 (Sb−O). ^{1}H NMR (DMSO-d_6, ppm): 6.96−7.96 (40H, m, Sb−C(Ph)), 8.39 (1H, d, J=3.1 Hz, C^6–H), 8.6 (1H, d, J=3.09 Hz, C^4–H). ^{13}C NMR (DMSO-d_6, ppm): 122.92 (1C, C−COOH), 125.79, 126.30, 128.68, 128.80, 129.38 (8C, Sb−C(Ph)), 131.51 (16C, *o*-carbons, Sb−C(Ph)), 135.53 (8C, *p*-carbons, Sb−C(Ph)), 132.53 (16C, *m*-carbons, Sb−C(Ph)), 137.82, 142.81 (2C, C−NO_2), 164.23 (1C, C−O), 166.68 (1C, C=O). Found, %: C 63.12, H 4.25. For $C_{62}H_{50}N_2O_7Sb_2$ calculated, %: C 63.13, H 4.24.

The photocatalytic activity of compound **1** was evaluated by the photodegradation of MB solution at room temperature. The photocatalytic reactions were carried out as follows: complex **1**·PhMe (25 mg) was dispersed in solution of MB (100 ml, aq., 6 mg · dm^{-3}) and placed under high pressure mercury UV lamp (125 W). Before turning on the lamp, the mixture was magnetically stirred in the dark for 30 min to reach an adsorption-desorption equilibrium. The change of MB concentration was controlled by UV spectroscopy according to the shift of the peak intensity (λ = 665 nm). A 5 ml aliquot of the reaction solution was periodically taken from the reactor, and dispersed powders were removed by centrifugation. The separated samples were analyzed by UV−Vis spectrophotometry. Degradation of the organic dyes under UV light without any complexes was also carried out for comparison.

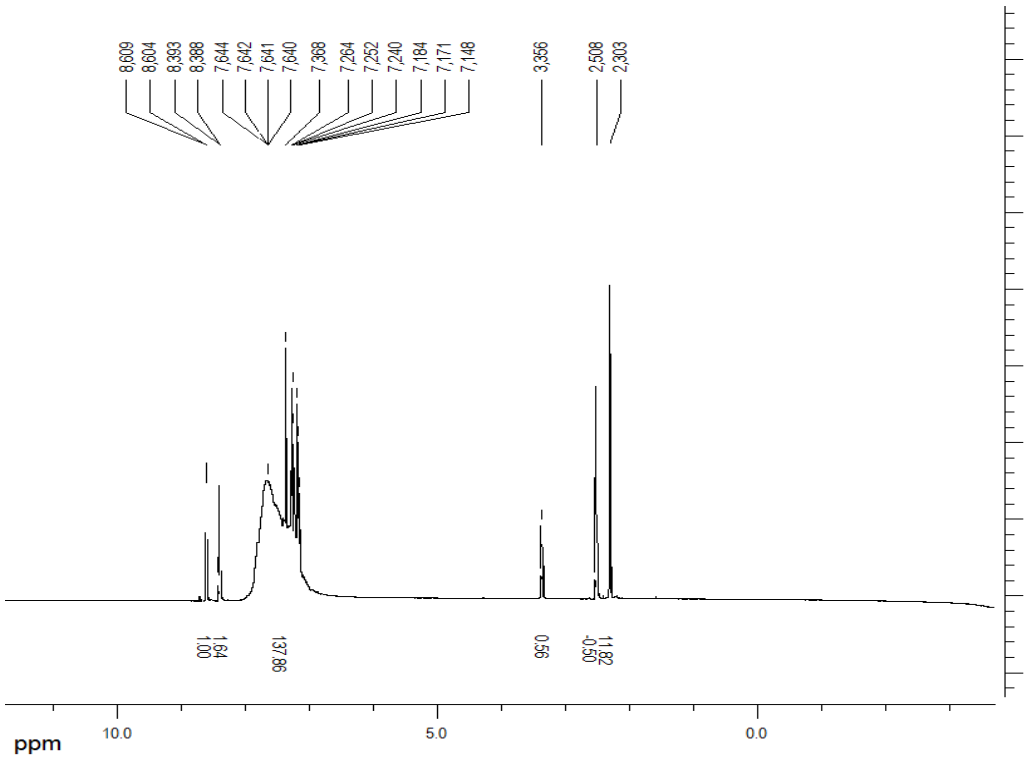

Figure S1. ^{1}H NMR of compound **1**·PhMe

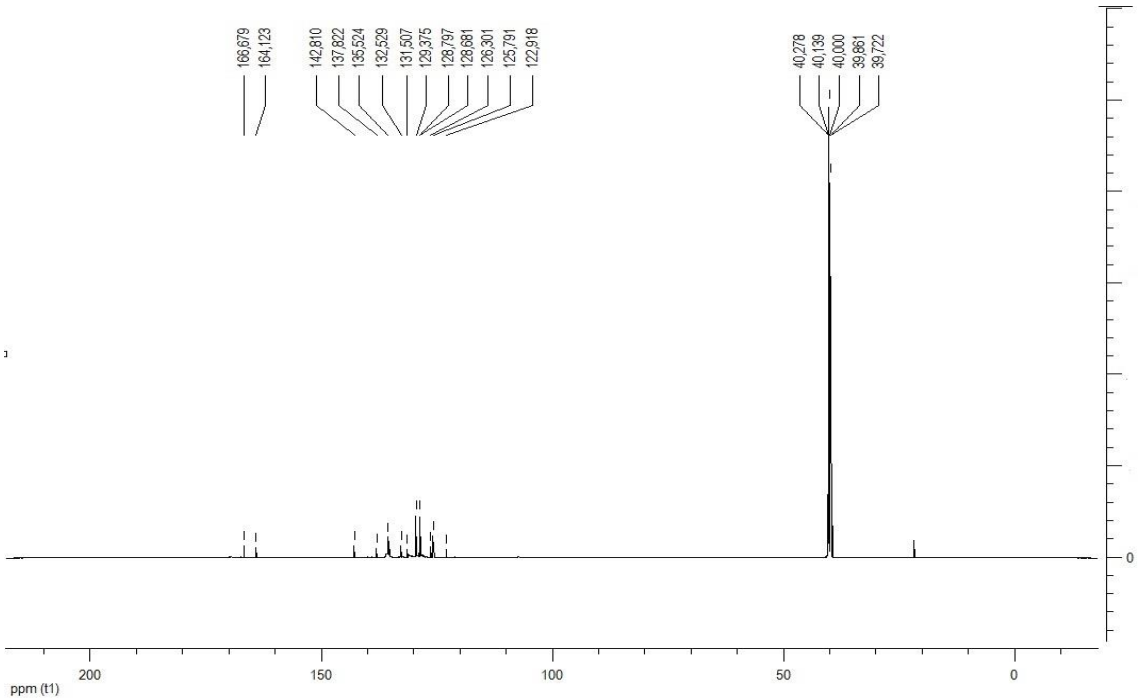

Figure S2. ^{13}C NMR of compound **1**·PhMe

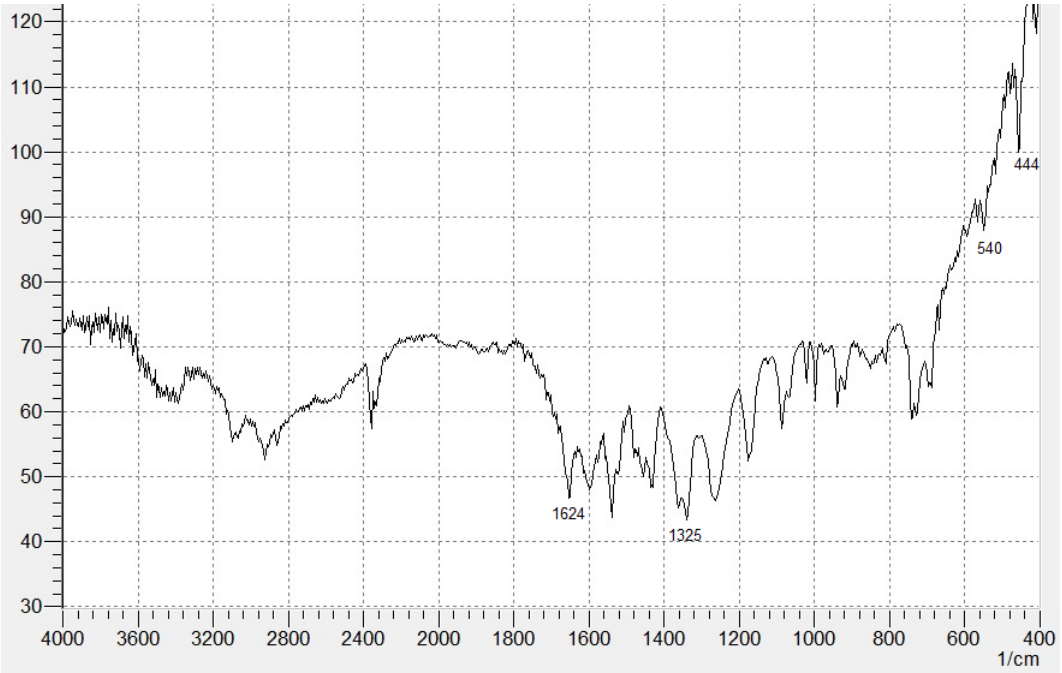

Figure S3. IR-spectrum of compound **1**

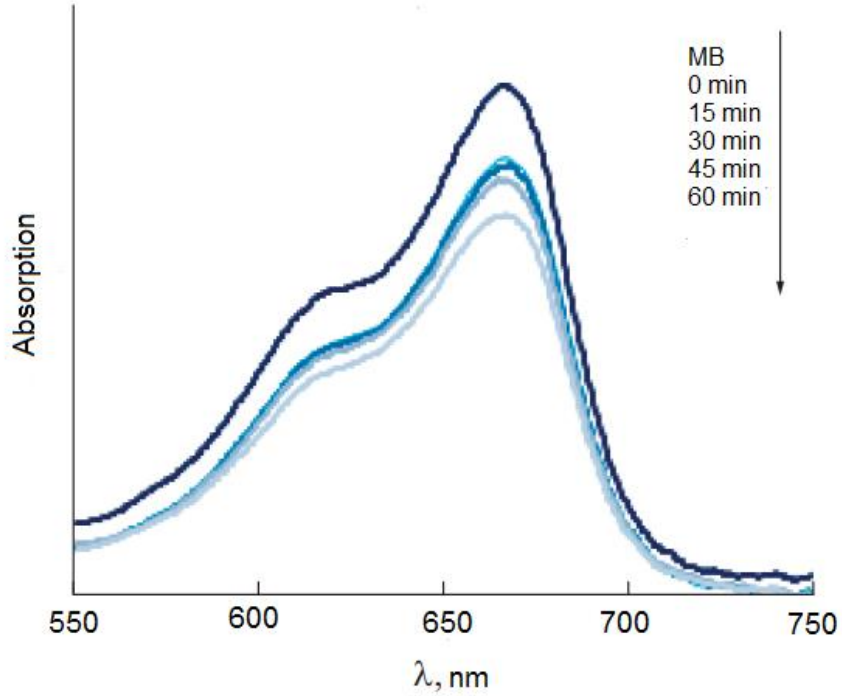

Figure S4. Absorption spectra of the MB solution in the presence of complex **1**