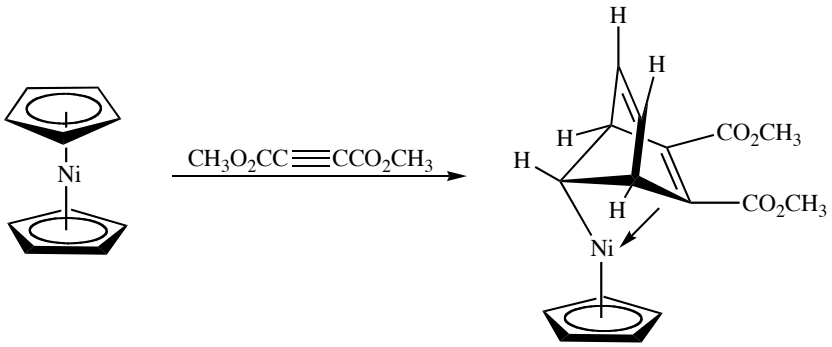
Results and Discussion (provided by SMM)

Nickelocene and dimethylacetylenedicarboxylate (DMAD) react to form the Diels-Alder adduct, bicyclo[2.2.1]heptadienylcyclopentadienyl nickel:



The product was recovered in 58.5% yield. This is a reasonable yield because, according to the literature, a 60-70 % yield is normally obtained after taking into account any unreacted nickelocene.[2] According to the literature, the melting point of nickelocene is 171-173 ˚C and that of the nickelocene/DMAD adduct is 84 ˚C or 85˚C.[2,3] My measured melting point range for the adduct is 83.2-85.4 ˚C, which is in agreement with the reported range, and therefore indicates the formation of the adduct.

The IR peaks for the adduct (Table 1) are similar to those found for nickelocene, except that C=O and C-O stretches for the ester group are present. The strong C=O stretch appears at 1674 cm^{-1}, C=C bands are at 1523, 1439 and 1403 cm^{-1}, and the C-O stretches are at 1301, 1242, and 1191 cm^{-1}. My ^{1}H NMR data for the adduct (Table 2) agree with the NMR data for the adduct reported in the literature[2] and indicate that the expected 18-electron diamagnetic product was obtained. It appears that one of the cyclopentadienyl rings in nickelocene reacted with DMAD to form a bicyclic ring that is bonded to nickel. The ^{1}H NMR spectrum shows five signals with 2:5:2:6:1 relative intensities. The signal around 6.6 ppm represents the two olefinic protons of the bicyclic ring. The singlet at 5.3 ppm represents the five equivalent protons on the cyclopentadienyl ligand. The signal at 3.8 ppm represents the two equivalent methine protons of the bicyclic ring. The singlet at 3.7 ppm represents the six protons of the methoxy group, and the signal at 2.3 ppm represents the lone proton bonded to the carbon with the free electron pair. Based on its IR data, diamagnetism, and NMR data, the product is the Diels-Alder adduct of nickelocene and DMAD and it has an electron count of 18 unlike nickelocene, which has an electron count of 20.

Table 1. IR data for the Nickelocene/DMAD Diels-Alder adduct as a nujol mull.

Assignment	**wavenumber (cm^{-1})**
Aromatic C-H	3091, 3024
Alkane C-H	2923
C=O	1674
C=C	1523, 1439, 1403
C-O ester	1301, 1242, 1191

Table 2. ^{1}H NMR data for the Nickelocene/DMAD Diels-Alder adduct in $CDCl_3$.

Assignment	chemical shift (ppm)	# of H
CH=CH	6.6	2
C_5H_5	5.3	5
=CCH	3.8	2
CH_3	3.7	6
:CH	2.3	1

(where brown is your conclusion)