

Supplementary Materials

Newly-Obtained Two Organic-Inorganic Hybrid Compounds Based on Potassium Peroxidomolybdate and Dicarboxypyridinic Acid: Structure Determination, Catalytic Properties, and Cytotoxic Effects of Eight Peroxidomolybdates in Colon and Hepatic Cancer Cells

Adrianna Sławińska ¹, Małgorzata Tyszką-Czochara ², Paweł Serda ³, Marcin Oszejca ³,
Małgorzata Ruggiero-Mikołajczyk ¹, Katarzyna Pamin ¹, Robert Karcz ¹ and Wiesław Łasocha ^{1,3,*}

- ¹ Jerzy Haber Institute of Catalysis and Surface Chemistry, Polish Academy of Sciences, Niezapominajek 8, 30-239 Krakow, Poland; adrianna.slawska@ikifp.edu.pl (A.S.); malgorzata.ruggiero-mikolajczyk@ikifp.edu.pl (M.R.-M.); katarzyna.pamin@ikifp.edu.pl (K.P.); robert.karcz@ikifp.edu.pl (R.K.)
² Faculty of Pharmacy, Jagiellonian University Medical College, Medyczna 9, 30-688 Krakow, Poland; malgorzata.tyszka-czochara@uj.edu.pl
³ Faculty of Chemistry, Jagiellonian University, Gronostajowa 2, 30-387 Krakow, Poland; pawel.serda@uj.edu.pl (P.S.); oszejcam@chemia.uj.edu.pl (M.O.)
* Correspondence: lasocha@chemia.uj.edu.pl

Register

1. Selected lengths of bonds for **K26dcpa** and **K35dcpa**.
2. Rietveld refinement plots for **K35dcpa**.
3. TG/DSC investigations for **K35dcpa** and **K26dcpa**
4. Catalysis for selected compounds comparing I, II, III run.
5. X-ray diffraction, IR spectroscopy, an elemental analysis for selected compounds *K-nicO*, *K-picO*, *NH₄-nicO*, **K35dcpa**, **K26dcpa** and *Na-picO* before and after BV reaction.

1. Selected lengths of bonds for K26dcpa and K35dcpa.

Table S1. Selected bond lengths (Å) in the studied compounds.

| K26dcpa | | K35dcpa | |
|----------|------------|---------|----------|
| Mo1 – O8 | 1.6898(17) | Mo1-O4 | 1.68(11) |
| Mo1 -O6 | 1.9176(24) | Mo1-O6 | 1.92(9) |
| Mo1 -O9 | 1.9239(25) | Mo1-O3 | 1.93(6) |
| Mo1-O12 | 1.9484(27) | Mo1-O5 | 2.20(9) |
| Mo1-O10 | 1.9813(28) | Mo1-O9 | 2.02(8) |
| Mo1-O4 | 2.0332(20) | Mo1-O10 | 1.99(6) |
| Mo1N1 | 2.4054(17) | Mo1-O3 | 1.93(6) |
| O4-O11 | 2.2286(27) | O6-O9 | 1.36(8) |
| O5-O7 | 2.2385(39) | O7-O3 | 1.34(11) |
| O6-O12 | 1.4728(32) | O18-O7 | 2.24(11) |
| C2-O5 | 1.2314(46) | C1-O1 | 1.28(7) |
| C2-O7 | 1.2806(43) | C1-O2 | 1.27(7) |
| C5-O11 | 1.2105(39) | O5-N1 | 1.33(8) |
| C5-O4 | 1.3101(30) | | |

2. Rietveld refinement plots for K35dcpa.

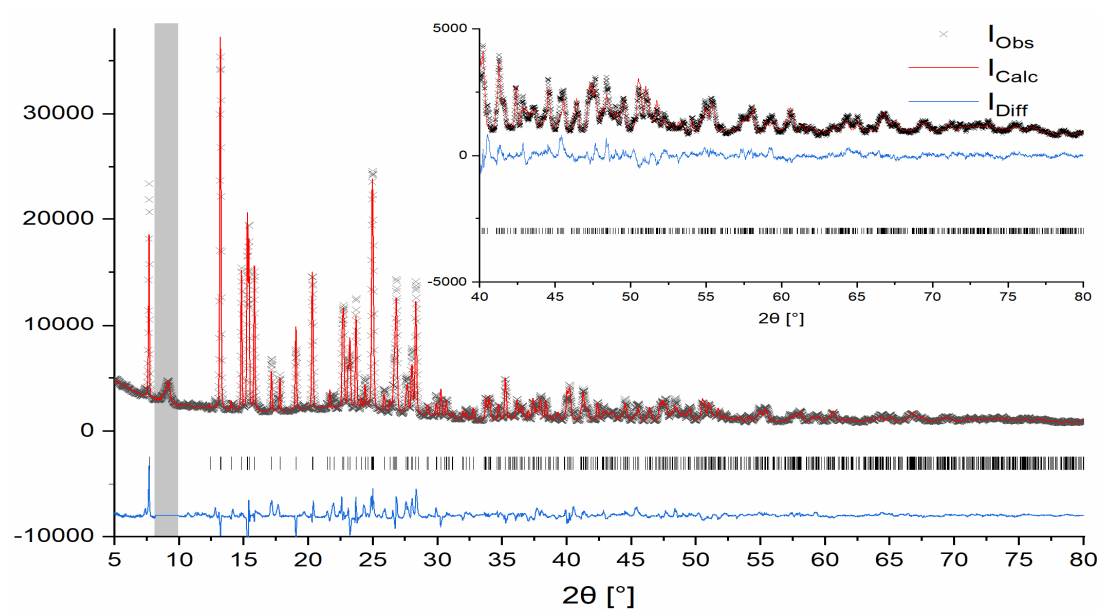


Figure S1. K35dcpa - Final Rietveld refinement plots [JANA2006 program]. Gray bar indicates the so-called 'excluded region' - excluded due to the presence of diffraction lines from the 'capillary system'.

3. TG/DSC investigations for K35dcpa and K26dcpa.

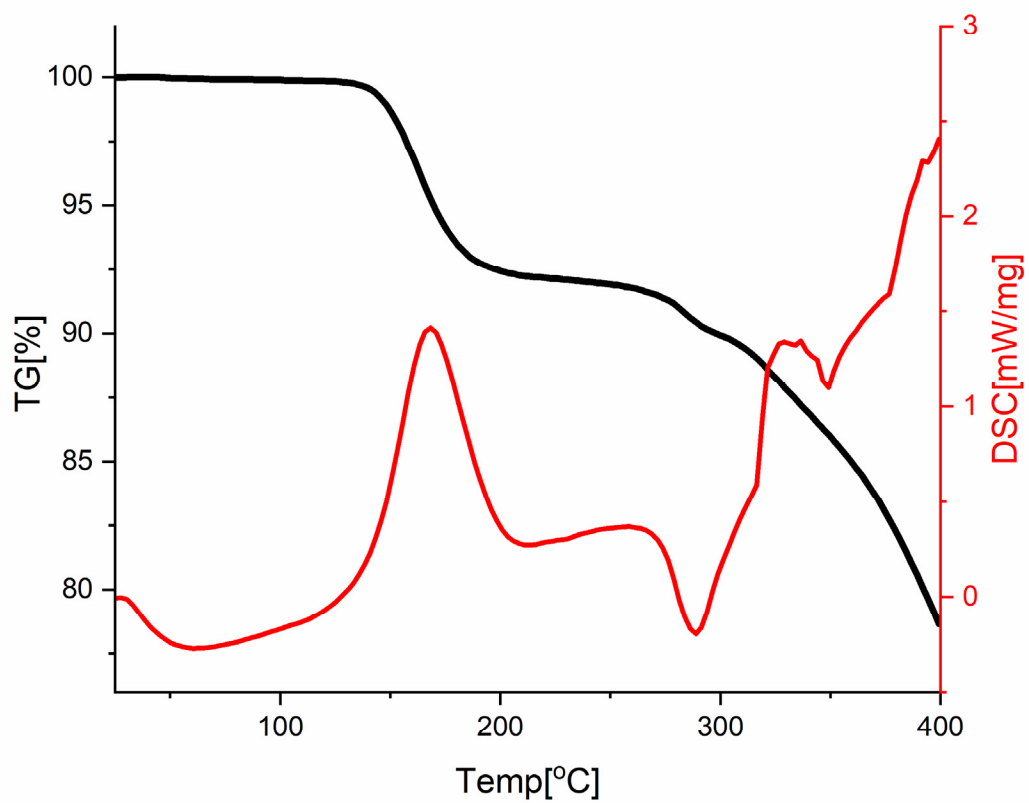


Figure S2. TG/DSC investigation for K26dcpa.

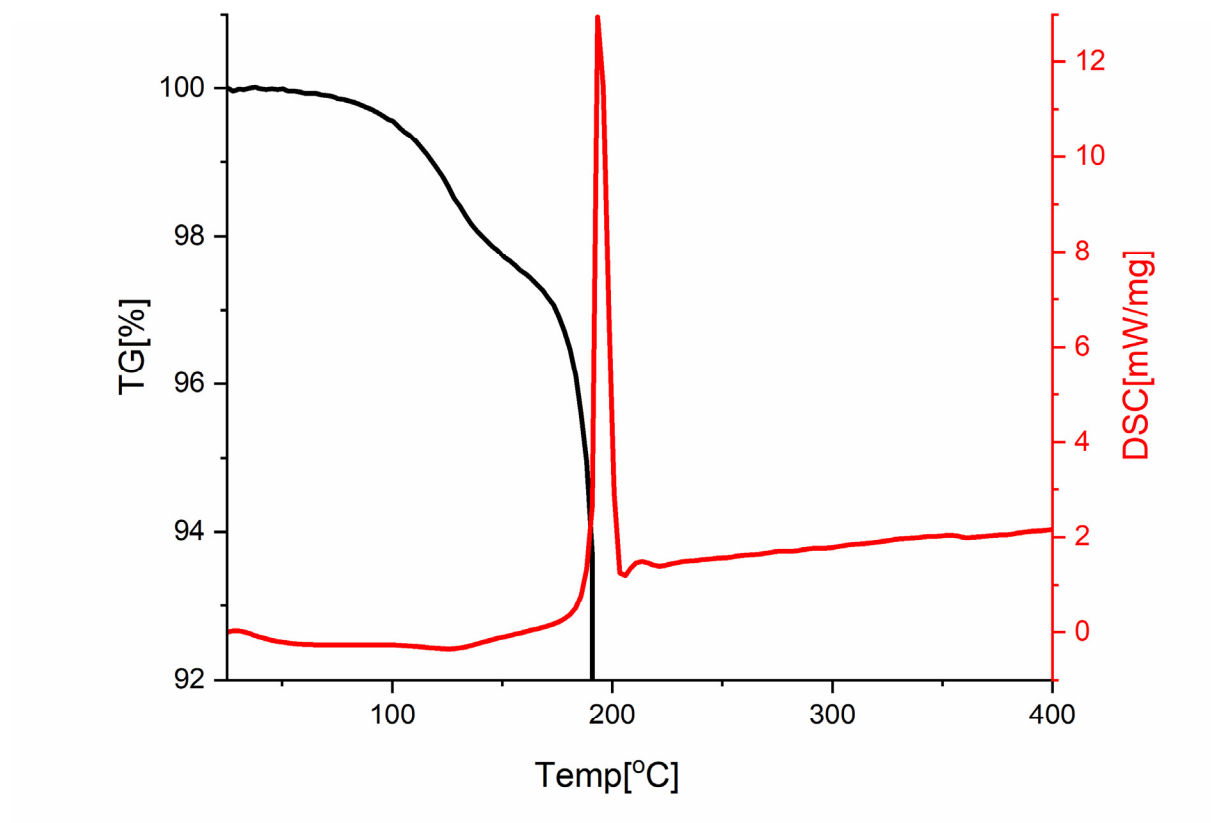


Figure S3. TG/DSC investigation for **K35dcpa**.

4. Catalysis for selected compounds comparing I, II, III run.

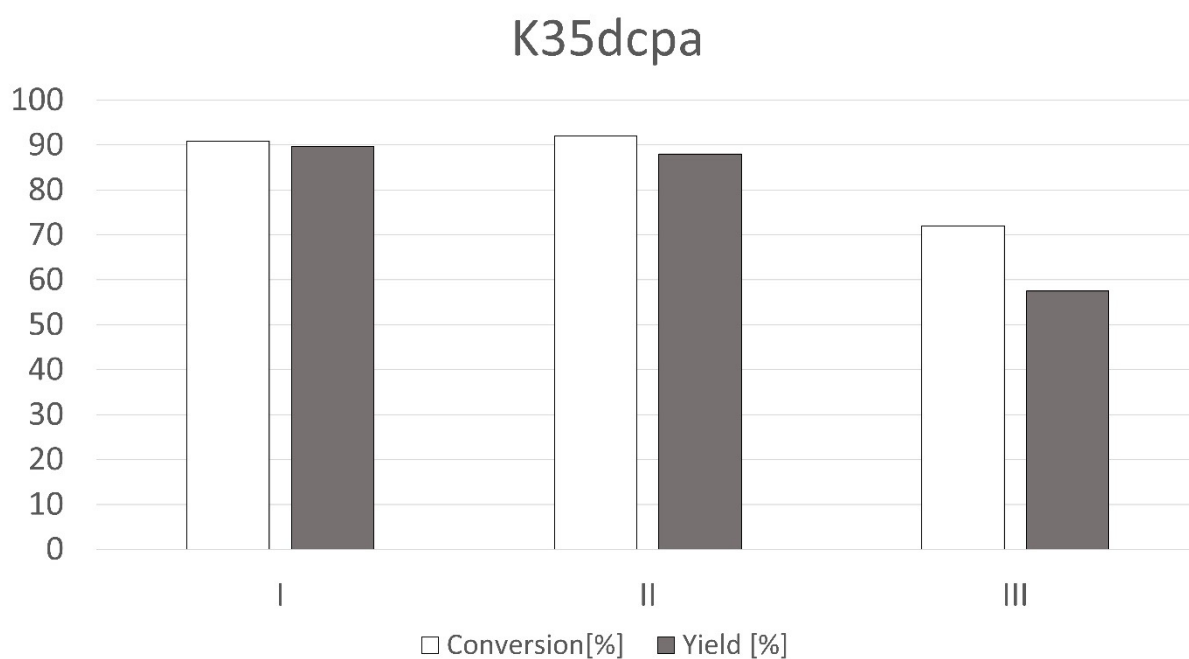


Figure S4. The BV oxidation of cyclohexanone with **K35dcpa** as catalyst in I, II, III run.

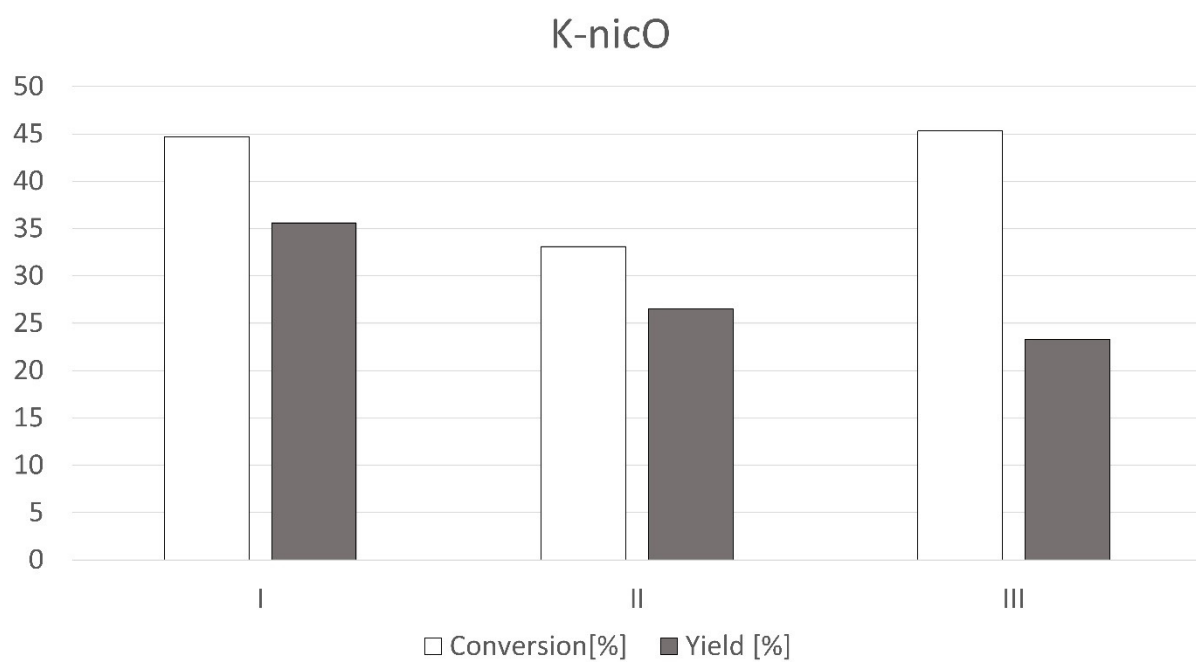


Figure S5. The BV oxidation of cyclohexanone with **K-nicO** as catalyst in I, II, III run.

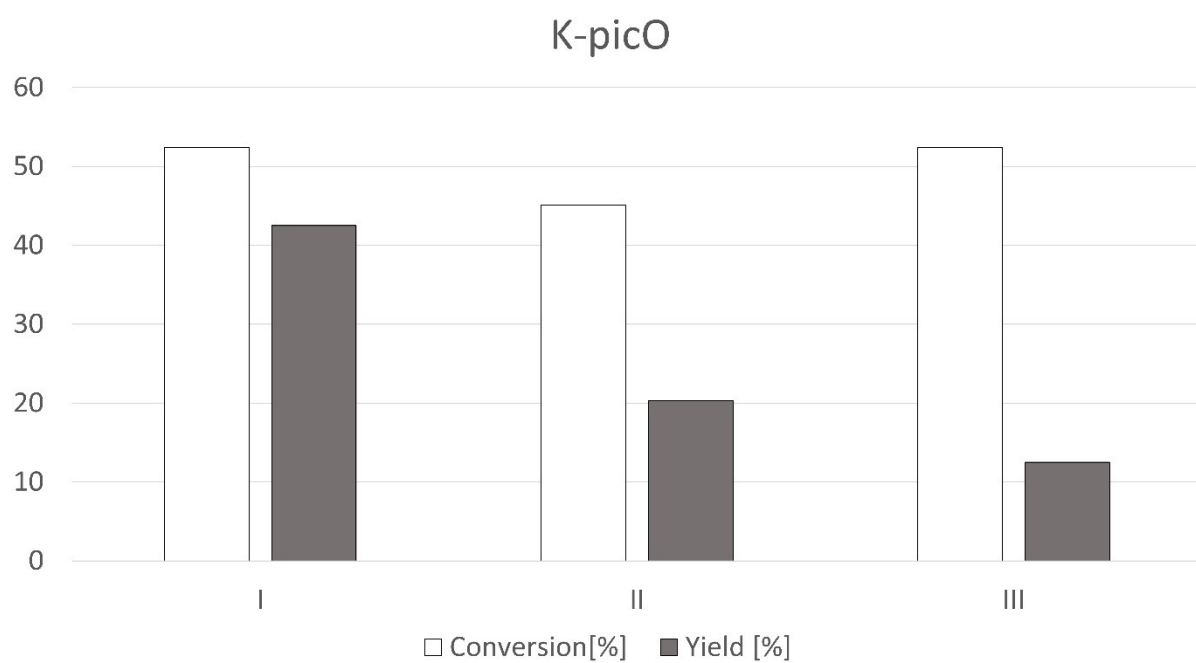


Figure S6. The BV oxidation of cyclohexanone with **K-picO** as catalyst in I, II, III run.

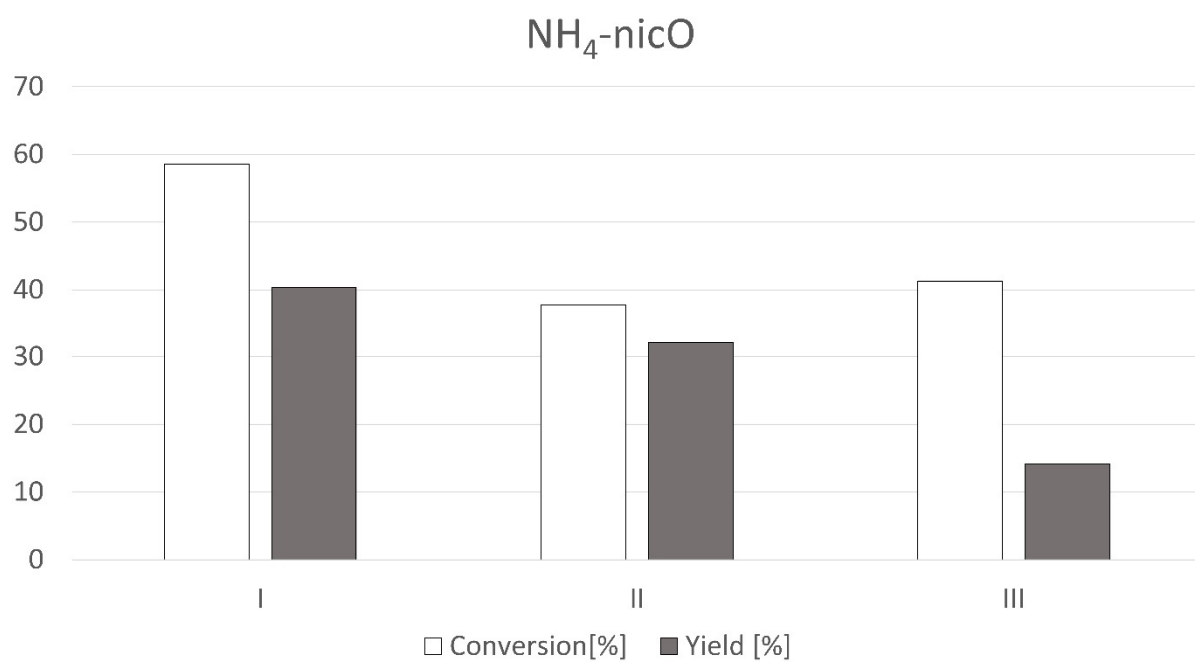


Figure S7. The BV oxidation of cyclohexanone with **NH₄-nicO** as catalyst in I, II, III run.

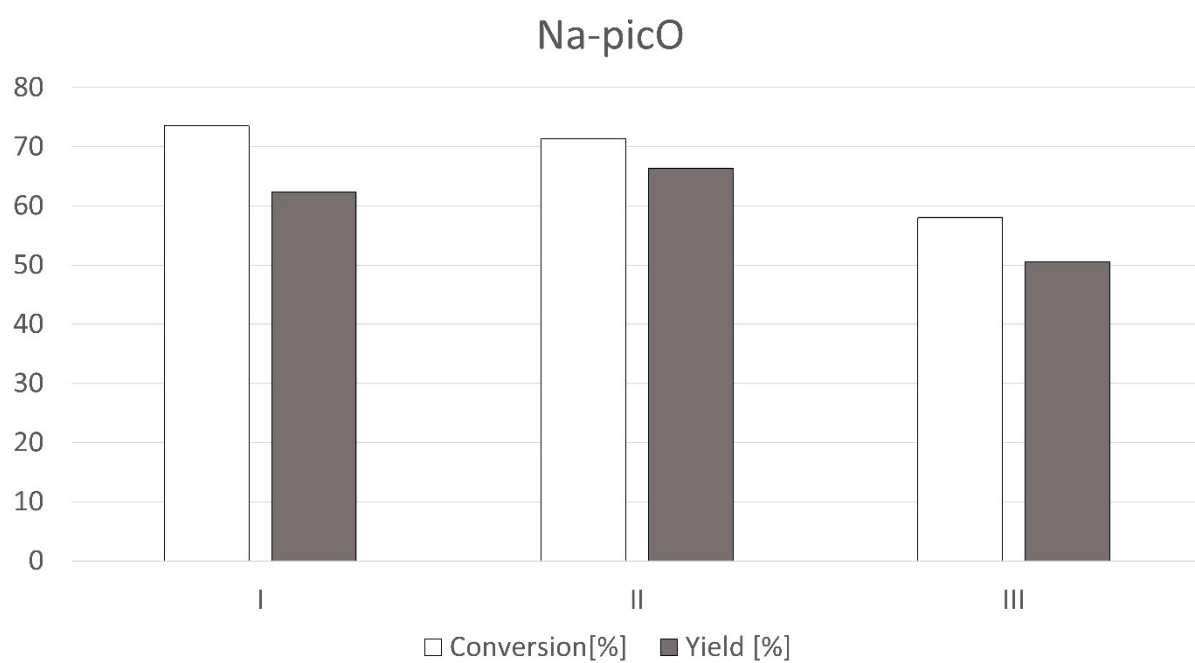


Figure S8. The BV oxidation of cyclohexanone with **Na-picO** as catalyst in I, II, III run.

5. X-ray diffraction, IR spectroscopy, an elemental analysis for selected compounds K-nicO, K-picO, NH₄-nicO, K35dcpa, K26dcpa and Na-picO before and after BV reaction.

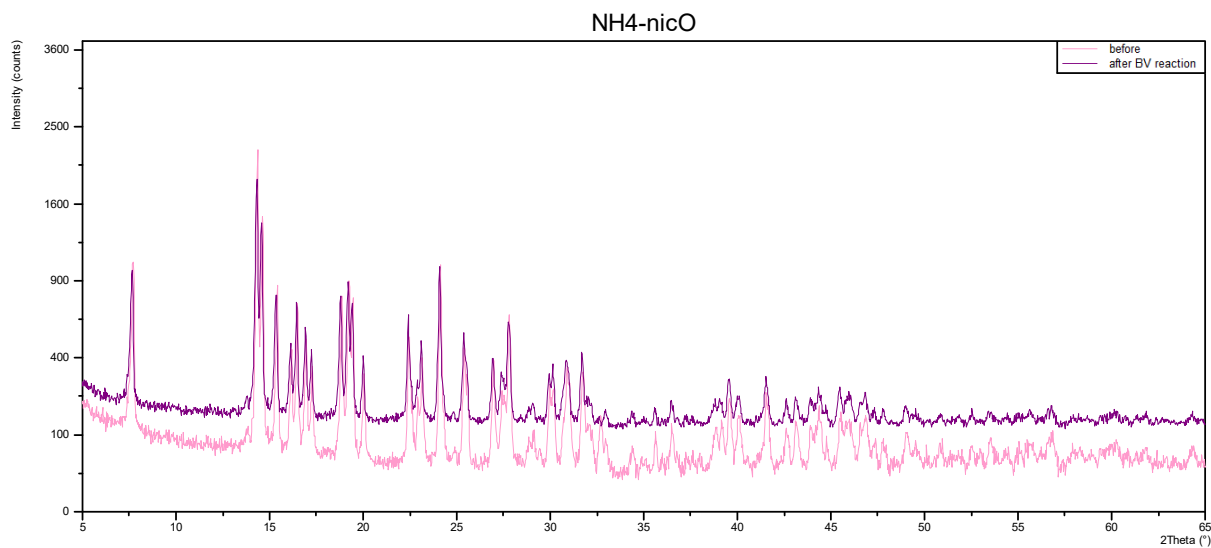


Figure S9. PXRD before and after BV reaction for NH₄-nicO.

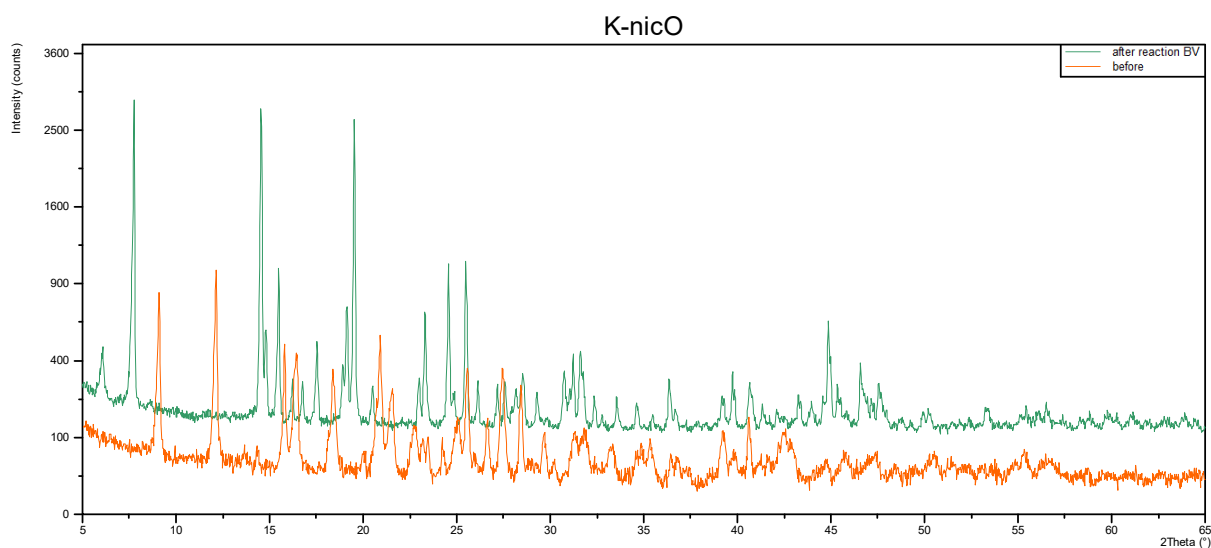


Figure S10. PXRD before and after BV reaction for K-nicO.

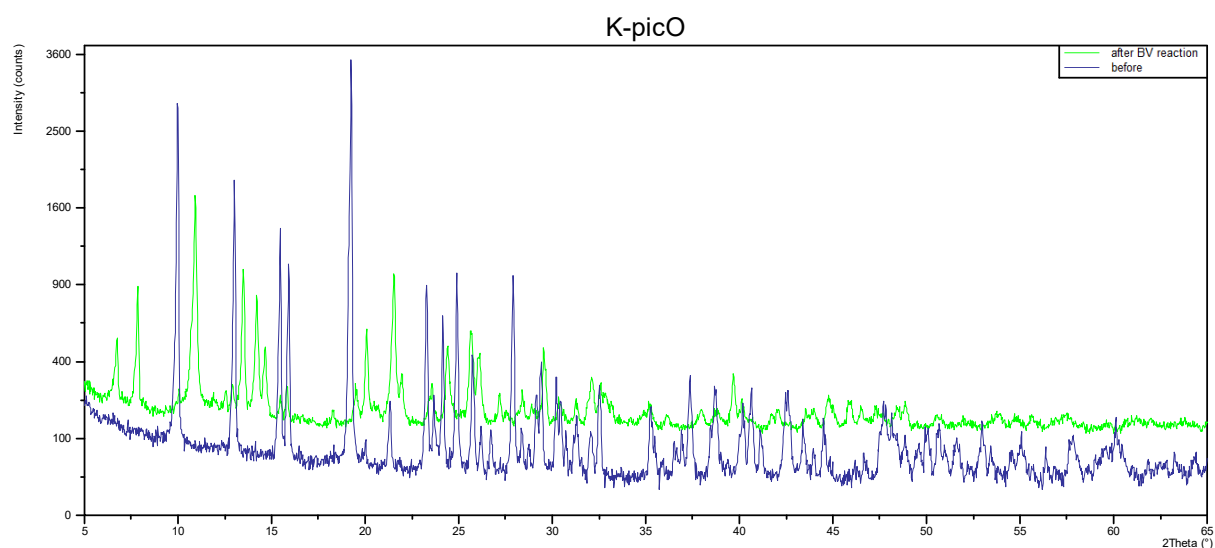


Figure S11. PXRD before and after BV reaction for K-picO.

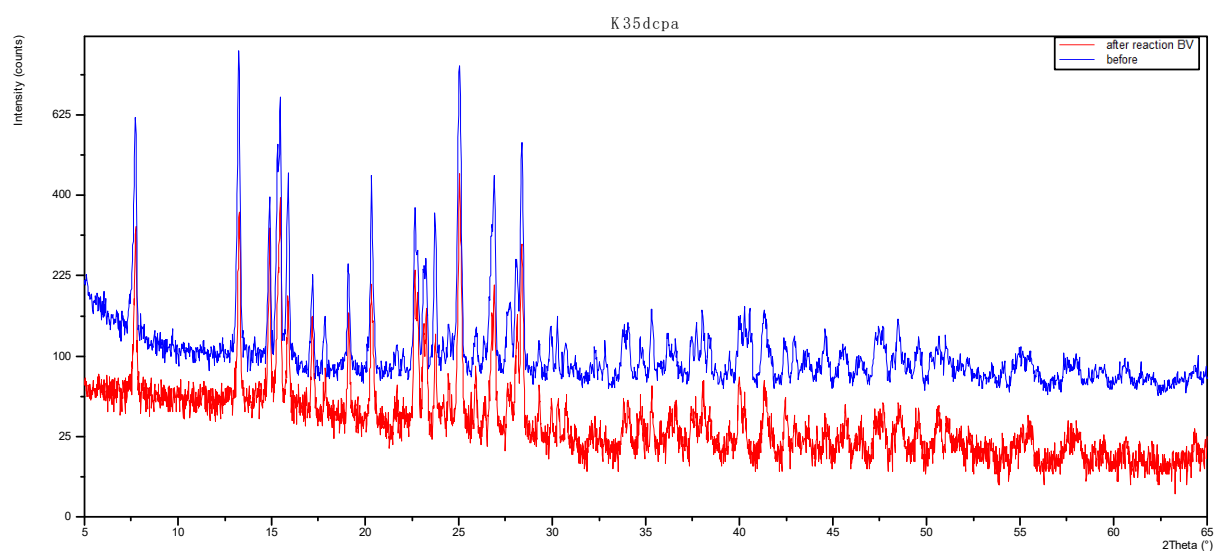


Figure S12. PXRD before and after BV reaction for K35dcpa.

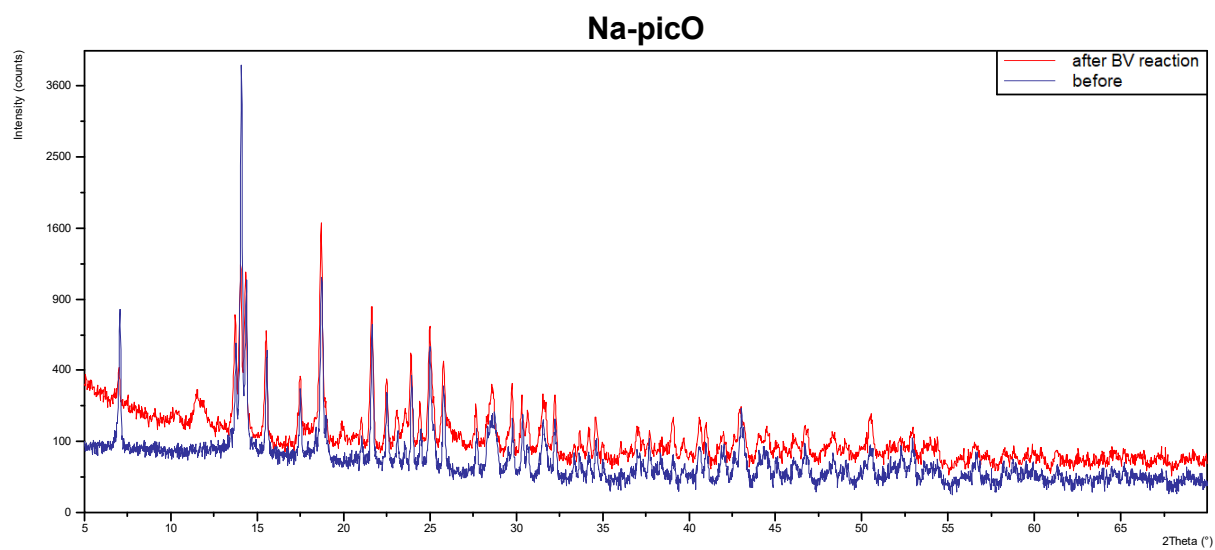


Figure S13. PXRD before and after BV reaction for Na-picO.

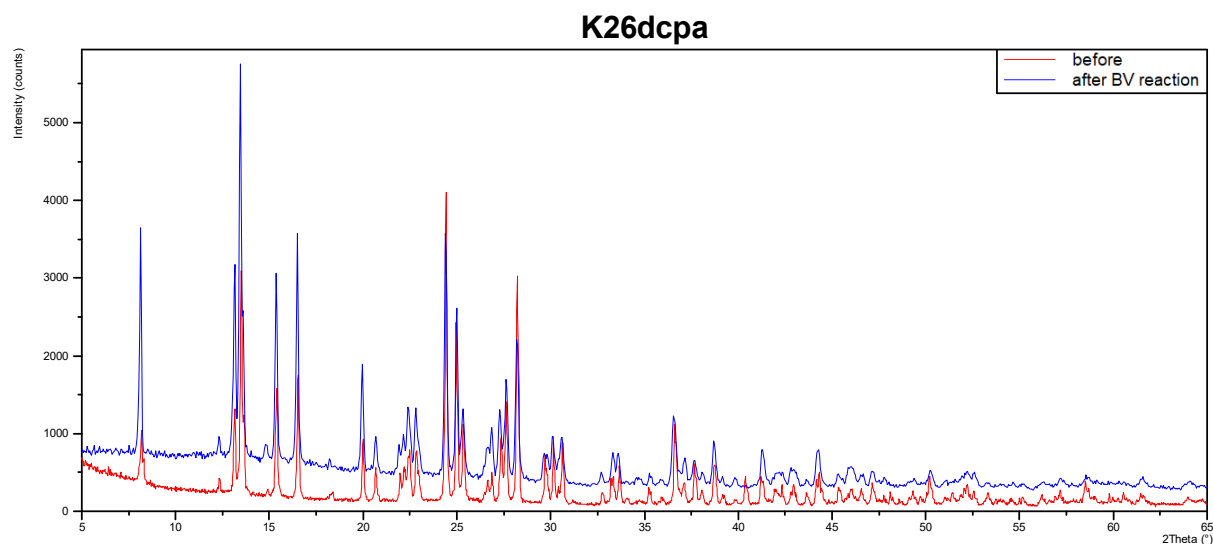


Figure S14. PXRD before and after BV reaction for **K26dcpa**.

Table S2. Result of elemental analysis for K-nicO and K-picO before and after BV reaction.

| K-nicO | | K-picO | |
|-----------------------|-------------------------------|-----------------------|---------|
| before | after (lost H ₂ O) | before | after |
| C 18.56 (calc. 19.42) | C 20.14 (calc. 20.41) | C 20.11 (calc. 20.41) | C 22.03 |
| N 3.6 (calc. 3.77) | N 3.96 (calc. 3.97) | N 3.9 (calc. 3.97) | N 4.27 |
| H 1.616 (calc. 1.63) | H 1.143 (calc. 1.14) | H 1.202 (calc. 1.14) | H 1.321 |

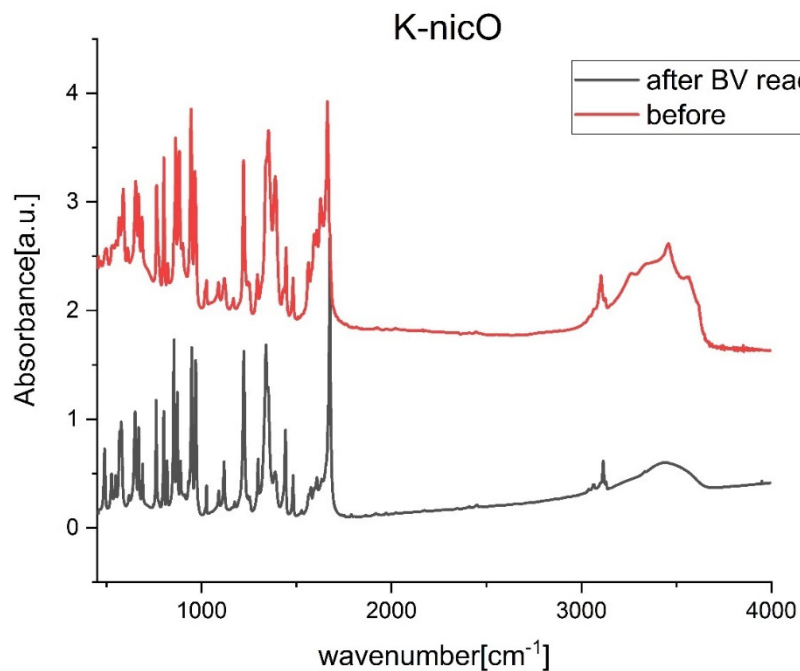


Figure S15. IR spectra of the compounds K-nicO before and after BV reaction.

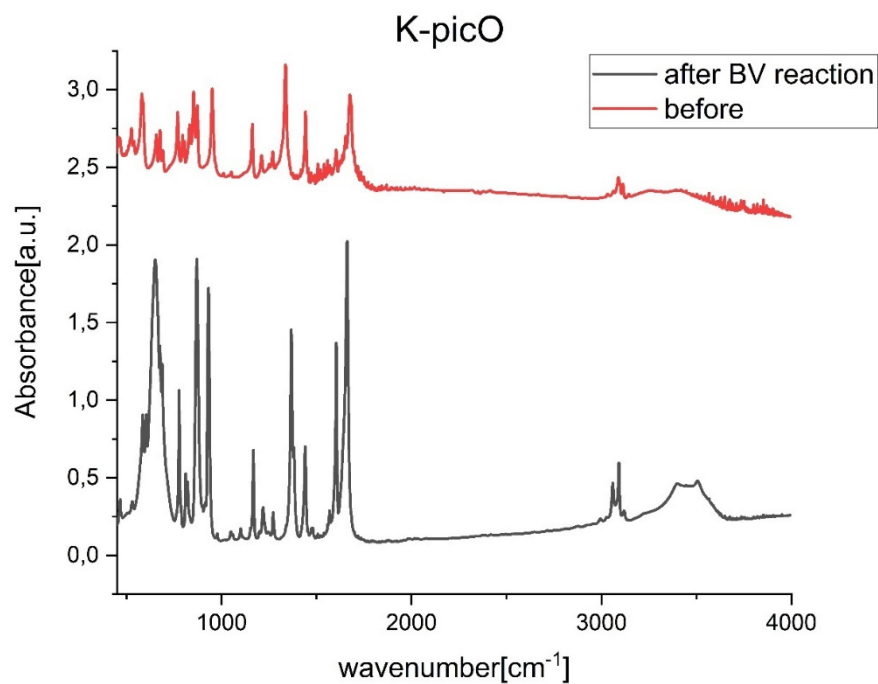


Figure S16. IR spectra of the compounds K-picO before and after BV reaction.

To conclude this part: Structure changes for *NH₄-nicO*, **K35dcpa**, **K26dcpa** and *Na-picO* were not observed, changes in XRPD patterns were observed in the case of *K-nicO* and *K-picO*. However, chemical analysis and IR spectra (recorded for these 2 compounds) indicate only small changes in 'structure skeletons'. Probably the mild BV reaction conditions causes mostly only dehydration in the catalyst.