anthelminthic preparations.

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STABILITY OF STATIONARY CATALYSTS FOR THE LOW-TEMPERATURE HYDROGENATION OF COTTONSEED MISCELLAS

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The stability of a catalyst is an important technical and economic index of the desirability of its use.

We have studied the stability of nickel–copper–aluminum catalysts with no additives and with the addition of 7.5% of molybdenum [1] in a semi-industrial apparatus of the column type installed at the Kokand oils and fats combine.

We performed hydrogenation by two methods: By the "jet" method the miscella and the hydrogen were fed into the bottom of the reactor, and in the "drop" method the miscella was fed at the top and the experiments were performed in a closed system without the circulation of hydrogen.

As the solvent we used extraction gasoline and its hexane fraction. On hydrogenation in extraction gasoline we used a miscella refined under industrial conditions (with a concentration of 52.3%), and model samples of hexane miscella (concentration 50.1%) were prepared from extraction oil.

The stability of the catalyst was studied under a constant pressure of hydrogen of 300 kPa at a hydrogenation temperature of 90°C with a space velocity of feed of miscella of 1 h^{-1} and a space velocity of feed of excess of hydrogen of 30 h^{-1}.

The results obtained indicate that the activity of nickel–copper–aluminum catalysts without additives falls less intensively when hexane is used as the solvent – by 34.9% on working for 500 h as compared with a 42% fall in activity on working for 306 h in the continuous hydrogenation of a gasoline miscella. This may be due to the lower amount of organosulfur compounds (0.001135%) in hexane as compared with extraction gasoline (0.002168%).

The study of the stability of a catalyst promoted with molybdenum showed that after 550 h of work the fall in activity was 10.5% (cottonseed oil with iodine number of 110.7% I_2 was hydrogenated in gasoline containing 0.001388% of organosulfur compounds).

Consequently, promotion of a nickel–copper–aluminum catalyst increases not only the activity [2] but also the stability of the catalyst, which is apparently connected with its resistance to catalyst poisons.

For regeneration, the catalysts studied, which had lost 77.2% and 50.1% of their activities (catalyst without additives and catalyst with the addition of molybdenum, respectively) were defatted with extraction gasoline and were then subjected to heating in a muffle furnace at 500-550°C in a current of atmospheric oxygen for 10-11 h, after which they were reduced at a temperature of 200-220°C under a pressure of hydrogen of 300 kPa for 3.5-4 h. The results of hydrogenation with a gasoline miscella showed that after regeneration by calcination the catalysts had practically regained their initial activity.

Thus, after the fall in activity as a result of the poisoning with sulfur compounds, stationary nickel–copper–aluminum catalysts without additives and with addition of a promotor (Mo) can be regenerated, thanks to which the useful life of a single portion of catalyst is increased.

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STUDY OF SOME REPRESENTATIVES OF THE FAMILY *Umbelliferae*

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By adsorption chromatography on neutral alumina, from the total extractive substances of the roots of *Seseli peucedamoides* K. Pol., *Lazer trilobum* Borkn., *Peucedanum cervarifolium* C.A.M., and *Peucedanum pauciradiatum* Tamamsch. we have isolated aromatic and coumarin compounds. On the basis of physicochemical characteristics, substances with the compositions $C_{11}H_{12}O_4$ (I), mp 85-86°C, from *Lazer trilobum*; $C_{12}H_8O_4$ (II), mp 188-191°C, from *Peucedanum cerevarifolium*; and $C_{24}H_{30}O_3$ (III), mp 61-63°C, from *Seseli peucedanoides* were identified as crocatone, bergapten, and umbelliprenin, respectively [1, 2].

In addition to crocatone and radiatinin [3], from the combined extractive substances of the roots of *Peucedanum pauciradiatum* we isolated a new aromatic compound not previously described in the literature, with the composition $C_{10}H_{10}O_4$ (IV), M$^+$ with m/z 194, mp. 169-170°C, which we have called radiatinol. The IR spectrum of (IV) has adsorption bands at (cm^{-1}) 3370 (hydroxylic OH), 1675 (CO of a conjugated ketone), 1630, 1615, 1530 (aromatic ring), and 1060 and 930 (methylenedioxy group). The ^{1}H NMR spectrum of radiatinol (Fig. 1) shows the signals of protons at (ppm) 1.13 (CH_3-CH_2-, 3 H, triplet, J = 7 Hz); 2.80 (CH_3-CH_2-, 2 H, quartet, J = 7 Hz); 5.95 ($-O-CH_2-O-$, 2 H, singlet); and 7.0 and 7.25 (hydrogen atoms of a benzene ring, 1 H each, doublets, J = 2.0 Hz). The size of the spin-spin coupling constant of the aromatic protons confirms their meta position [4]. The methylation of radiatinol with diazomethane formed crocatone [1]. The positions of the substituents in crocatone were confirmed by the Overhauser effect. Thus, irradiation of the protons of the methoxy group by a H_2 field led to a rise in the intensity of the signals of the signal from the H-3 proton by 20%, while the intensity of that from the H-1 proton did not change.

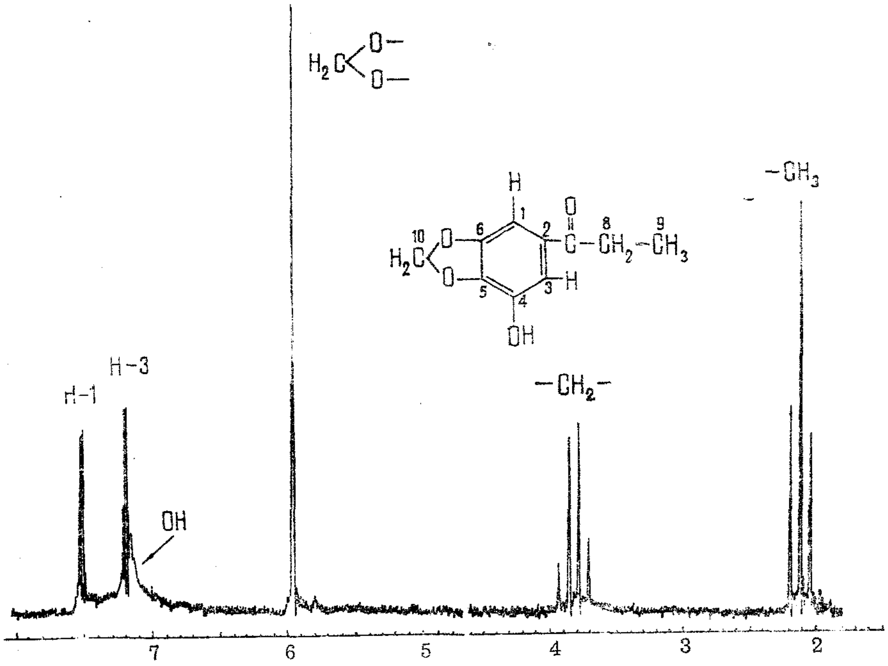

Fig. 1. ^{1}H NMR spectrum of radiatinol.

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