

## Infrared Bands of OH Groups in HY Zeolite. A Solvent Effect in Sample Preparation for ir Studies

The most frequently applied technique in ir studies of OH groups in HY zeolite is the use of a pressed disk. Dehydration and de-ammoniation of the self-supporting disk of  $\text{NH}_4\text{Y}$  is then commonly carried out under conditions analogous to those designated by Kerr (1) as "shallow bed" conditions, e.g., pressure  $< 0.1$  Torr,  $T = 350^\circ\text{C}$ , thickness of the sample 0.5 mm (ca. 5-10 mg/cm<sup>2</sup>), decomposition time 3-4 hr. There are then bands in the OH stretching region at 3750, 3640 and 3550 cm<sup>-1</sup>. The bands at 3640 and 3550 cm<sup>-1</sup> correspond to the valency vibrations of the structural OH groups of HY, and it is assumed that the band at 3750 cm<sup>-1</sup> is due to terminal silanol groups or silica gel like impurities.

After hydrating such a calcined sample of zeolite Y [in our case with a composition  $0.29 \text{ Na}_2\text{O} \cdot 0.033 \text{ CaO} \cdot 0.72 (\text{NH}_4)_2\text{O} \cdot \text{Al}_2\text{O}_3 \cdot 4.95 \text{ SiO}_2$  (about 30% of the original content of  $\text{Na}^+$ )] and repeating the dehydration under the same conditions, one obtains a spectrum (Fig. 1) which shows a marked rise in the band at 3750 cm<sup>-1</sup>. The unwanted changes of the spectra caused by the rehydration in the preparation of the self-supporting pellet were removed by us by applying the Nujol suspension technique and using a dry box. The resulting spectra showed in the hydroxyl stretching region only one band with a maximum at 3550 cm<sup>-1</sup>. The spectrum corresponds with the spectrum obtained by Venuto, Wu and Cattanaach (2) under the same conditions.

By using a polyphenyl ether (Convalex, supplied by Leybold-Heraneus) as the liquid medium for preparation of the ir specimen, a spectrum was obtained with three bands in the OH stretching region at 3740, 3640, and 3550 cm<sup>-1</sup>. The HY spectra

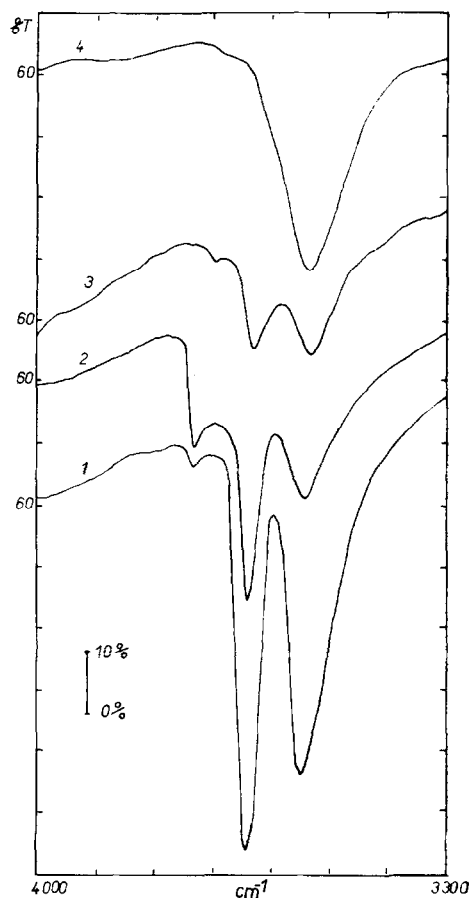


FIG. 1. (1) Hydroxyl stretching region of (H, Na) Y zeolite (30% Na<sup>+</sup>) for a sample pelleted and then calcined in vacuum for 3.5 hr at 350°C; [(NH<sub>4</sub>, Na)X → (H, Na)X]; (2) as (1), but hydrated in air after the first calcination, then again calcined in vacuum for 3.5 hr at 350°C; (3) hydroxyl stretching region of HNaY zeolite (30% Na<sup>+</sup>) for a sample calcined as a powder in vacuum for 3.5 hr at 350°C [(NH<sub>4</sub>, Na)Y → (H, Na)Y] and then the suspension prepared in Convalex; (4) as (3), but with the suspension prepared in Nujol. [NB. Convalex and Nujol show no absorption in the range 3200-4000 cm<sup>-1</sup>.]

from samples prepared for the ir measurement in the dry box (a) in the Nujol, (b) in Convalex, are also shown in Fig. 1. The shift of the absorption band at  $3640\text{ cm}^{-1}$  toward lower frequencies is ascribed to the interaction of the OH groups with the *n*-alkanes of the commercial Nujol. In contrast to the linear chains of the molecules of *n*-alkanes, the molecules of the polyphenyl ethers are bulky, and for steric reasons are not capable of interacting with the structural OH groups in the zeolite framework. The proof is a spectrum which does not show any change in the hydroxyl stretching region.

The results show that it is not appropriate to use Nujol in the suspension ir technique if one is interested in following

the changes generated in the hydroxyl stretching region of decationized Y zeolites.

#### REFERENCES

1. KERR, G. T., *J. Catal.* **15**, 200 (1969).
2. VENUTO, P. B., WU, E. L., AND CATTANACH, J., Conference on Molecular Sieves, London 1967. Ed: Society of Chemical Industry, London, 1968.

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