

Preparation of Stoichiometric Phosphates and the Effect of pH

Vogel and Marcelin (1) have commented on the preparation of stoichiometric aluminum phosphate particularly in reference to the work of Itoh, Tada, and Hattori (2) and that from this laboratory (3). The primary concern of Vogel and Marcelin is with the pH dependence of the preparation.

The present authors recognize, with Vogel and Marcelin (1), the importance of pH in preparations of catalysts. In our earlier publication (3) referred to above, the description of the preparation methods for the various stoichiometric phosphates was abbreviated in order to save journal space. Relatively few details of the preparation method were given, except to note the materials which were employed. Given the present opportunity, the authors wish to provide further information on the preparation methods used and the results obtained. More complete details are available elsewhere (4).

In the first method (method 1 of (3)) an aqueous solution of disodium hydrogen phosphate was combined under vigorous agitation with a solution of a simple salt of the metal cation. The precipitate obtained was settled, filtered, and extensively

washed with water. It was then dried and sieved, and the fraction of 60–150 mesh was retained.

In the second method (method 2 of (3)), an aqueous solution of a salt of the metal cation was mixed with the stoichiometric amount of phosphoric acid. Concentrated ammonium hydroxide was then added slowly dropwise with continuous stirring until a pH was reached at which precipitation occurred. Such precipitates were treated further as in method 1.

In the preparation of the stoichiometric phosphates of the metals reported earlier (3), the pH at which precipitation occurred in method 2 was found to differ with the particular cation involved. These are summarized in Table 1.

As is readily observed the pH values at the precipitation "endpoint" all fall in the range from 4–6. Vogel and Marcelin (1) report that a pH of 4.0 or less produces pure aluminum phosphate, while a pH of 8.5 produces an amorphous X-ray pattern. Our value of 5 for the pH in the preparation of aluminum phosphate appears to agree reasonably well with that of these authors.

REFERENCES

1. Vogel, R. F., and Marcelin, G., *J. Catal.* **80**, 492 (1983).
2. Itoh, H., Tada, A., and Hattori, H., *J. Catal.* **76**, 235 (1982).
3. Gallace, B., and Moffat, J. B., *J. Catal.* **76**, 182 (1982).
4. Gallace, B., M.Sc. thesis, University of Waterloo, 1981.

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TABLE 1

pH Values at the
Precipitation "Endpoint"
in the Preparation of
Phosphates

Phosphate	pH
AlPO ₄	5
Ca ₃ (PO ₄) ₂	6
CrPO ₄	6
Co ₃ (PO ₄) ₂	4
Ni ₃ (PO ₄) ₂	5
FePO ₄	6