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Reply to the comment by Bailey and Waugh on the use of temperature programmed desorption of H_2 to determine metal surface area of Cu catalysts

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In the comment [1] on our recent letter on metal area determination of copper catalysts by use of H_2 TPD [2], Bailey and Waugh express their reservations to the use of this technique. They base their critique on the fact that they fail to obtain well-resolved H_2 TPD peaks.

We agree with the authors of ref. [1] that their TPD results are not well suited for copper area determination and assume that the difficulties experienced by the authors are related to their choice of experimental conditions which apparently are different from those used by us [2]. Therefore, we advise Bailey and Waugh to employ extreme caution in making general conclusions regarding the adequacy of the H_2 TPD method. It is quite clear that the fact that they have succeeded in finding experimental conditions which give ill-resolved peaks cannot be used as an argument against the H_2 TPD method per se. In fact, in ref. [2] we report that well-resolved, reproducible peaks originating from desorption from copper metal can be obtained with samples similar to those studied in ref. [1]. In addition, Roberts and Griffin [3], and very recently Berndt et al. [4] have indeed also succeeded in obtaining good quality H_2 TPD data.

With the limited experimental details given by Bailey and Waugh it is of course difficult to speculate on the exact reasons why the authors have only succeeded to obtain poorly resolved desorption peaks. One reason may be non-linearity in the heating ramp as suggested by the apparent non-linear temperature scale. Other possible explanations could be related to the large sample sizes (about four times the amount that was used in our study), temperature gradients, diffusional limitations, etc. In any case, we recommend to carry out H_2 TPD using the same conditions as those in ref. [2].

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Regarding N_2O frontal chromatography we agree that it is a valuable method which many researchers including ourselves have had much use of. However, in ref. [2] we wanted to point out that it does have some inherent drawbacks. The method may be destructive, and as shown previously by many authors it may be difficult to find generally valid experimental conditions where no bulk oxidation takes place. Clearly, the H_2 TPD technique is advantageous in these respects.

In their discussion on which of the two methods give the correct copper area Bailey and Waugh base their arguments on *postulating* that the N_2O method gives the correct value. We believe this is a case of circular argumentation.

In a forthcoming publication we will compare in more detail N_2O frontal chromatography with hydrogen TPD and other methods [5], as well as discuss additional information which can be gained from H_2 TPD.

References

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