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Mepivacaine Hydrochloride Polymorphs: a Drug Production Quandary
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The existence of different crystalline forms of Mepivacaine.HCl was revealed by routine quality controls of industrial batches.

Two polymorphic non-solvated modifications were identified and characterized by powder diffraction data: Form II, the commercial one, accepted as a standard, and Form I, the more stable one, obtained by re-crystallization.

Single-crystal structure determinations of ethanol and methanol solvates, respectively Form III (possible precursor of industrial processing) and Form IV, have been carried out [1]. Combined with other data, these have shed light on possible interconversions between some of the above forms. In particular, microcrystalline Form II should be generated by desolvation of Form III in the course of industrial crystallization from ethanol and Form I may then be reached, in suitable conditions, *via* modest structural rearrangements.

[1] Giannellini V., Bambagiotti-Alberti M., Bartolucci G., Bruni B., Coran S.A., Costantino F., Di Vaira M., *J. Pharm. Biomed. Anal.*, 2005, *submitted*.
Keywords: mepivacaine-hydrochloride, polymorphs, X-ray diffraction

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Retarding Effects of Cellulose Ethers on Early Portland Cement Hydration
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Cellulose ethers (CEs) have phase-specific retarding effects on cement hydration. The retardation is correlated with the CE-adsorption on the mineral surfaces and depends on the CE´s degree of substitution (DS). The lower the DS the higher the amount of adsorbed CE and the stronger the retardation. The following phase reactions have been monitored in situ using synchrotron X-ray powder diffraction with a fast multi-strip detector:

1. $C_3A + C\overline{S} / C\overline{S}_{0.5} + H \rightarrow$Aft ($1^{st}$ ettringite formation)
2. $C\overline{S} / C\overline{S}_{0.5} + H \rightarrow C\overline{S}_2$ (gypsum crystallization)
3. $C_3A + C\overline{S}_2 + H \rightarrow$ AFt (2^{nd} ettringite formation)
4. $C_3S/C_2S + H \rightarrow CSH + CH$ (portlandite precipitation)

The C_3S-hydration is strongly and DS-specifically retarded. The retardation of gypsum crystallization is much weaker but distinguished, depending on DS. The first ettringite formation is not affected by CE. This is correlated with the adsorption rates, e.g. for a CE with a DS of 1.7: 540 µg/m² on portlandite, 240 µg/m² on gypsum and no adsorption on ettringite. The second ettringite formation does not fit into the scheme. It is strongly DS-dependently retarded although no CE adsorbs on ettringite. In the presence of silicates the second ettringite formation always takes place simultaneously with portlandite precipitation. The kinetics of the second ettringite formation in cement might therefore be influenced by the Ca-concentration in the pore water. The hydration of pure phase mixtures of C_3A and sulfates without silicates was studied in experiments using water and $Ca(OH)_2$-saturated solution. The ettringite crystallization is strongly inhibited in $Ca(OH)_2$-saturated solution.
Keywords: in-situ cement hydration, synchrotron powder diffraction, microstrip detector

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***In situ* Studies of Model High-Temperature Shift Catalysts**
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High-temperature shift (HTS) catalysts are used to catalyze the water-gas shift reaction in which CO with steam is converted to CO_2 and H_2. This reaction is an intermediate process in the industrial production of hydrogen used for e.g. the synthesis of ammonia. The product gas from a steam reformer is cooled in two processes: a high-temperature (320-500°C) step over a Cr containing magnetite catalyst and a low-temperature step (~220°C) over a Cu-Al_2O_3-ZnO based catalyst that reduces the CO concentration to about 0.1%.

In this combination of in situ studies, model HTS catalysts were prepared in order to determine the structure of pure and Cr containing iron oxides under industrially relevant conditions. From XRD it was observed that the addition of Cr resulted in lower crystallite sizes of the activated magnetite catalysts. Quantitative phase analysis using Rietveld refinement, the Avrami- and Arrhenius expressions, resulted in activation energies for the reduction process. XAFS, at the Cr- and Fe K-edges, showed the short-range order and oxidation states. TEM images illustrated that elongated particles become more dominant with increasing Cr concentration for the chloride-based synthesis. The sizes are generally in good agreement with XRD results. STEM studies combined with EDS suggested that the Cr concentration at the surface of the reduced particles was enhanced. Finally, electron diffraction showed transformations of crystallographic axes from Cr containing hematite particles to Cr containing magnetite particles.
Keywords: high-temperature shift catalysts, in situ structure determination, XRD XAFS TEM