# REPORT

Experiment **CH-3696**

"Structural characterization of novel metal-organic frameworks (MOFs), metal-organic coordination polymers (MOCPs), and host-guest MOF-derived materials"

In the framework of experiment **CH-3696** granted by ESRF for 6 shifts on beam line ID31, two scientists from Moscow (Russian Federation), namely, Dr. Vera I. Isaeva and Dr. Vladimir V. Chernyshev, have spent five days in Grenoble in the end of November, 2012. During 6 shifts - started on 28 November at 08:00 and ended on 30 November at 08:00 - thirty (30) high-resolution powder patterns of 30 compounds were measured at room temperature at beam line ID31. The X-ray wavelength used was 0.399810(13) Å. The patterns were measured in the 2θ ranges 0.2 – 25 or 0.2 – $30^o$. The Figures of all 30 patterns are given below (in the 2θ range 0.5 – $20^o$).

Samples **1** – **5** are representatives of the novel heteroaromatic MOFs obtained from $Zn^{2+}$ and pyridine2,5dicarboxylic/pyrazine2,5dicarboxylic acids. The careful analysis of the patterns have shown that each of these compounds contains two crystalline phases. However, thanks to high-resolution synchrotron experiment, four patterns were successfully indexed as two-phases ones. The crystal structures of all found phases were solved and refined. The rest pattern **3** is still in progress with indexing, nevertheless, the paper covering compounds **1** – **5** is in preparation.

Samples **6** – **21** are representatives of the host-guest MOF-based materials with various calix[4]arene derivatives introduced into MOF-5 cavities during the synthesis. Some of these compounds are not stable at ambient conditions, unfortunately. So patterns **15**, **18** and **21** (see Figures below) clearly show that these three compounds decomposed (within three months after the synthesis, when they provided acceptable powder patterns). All the rest patterns will be processed as soon as the structural work with the compounds **1** – **5** will be finished.

Due to robotization of the sample-change procedure on ID31 and kind assistance of Andy Fitch, who helped us in optimization of data collection strategy, nine extra samples **22** – **30** were measured before ending of our experimental time. These additional samples are the compounds with the unknown crystal structures, which can not be solved by us for a long time based on the laboratory powder patterns only. As a first result of the synchrotron measurements on ID31, the crystal structure of compound **22**, which is a new derivative of 1,2,4,5,7,8-hexaoxa-3-silonane, has been solved (see Figure 1), and the corresponding paper has been prepared to be submitted in *Organometallics*.

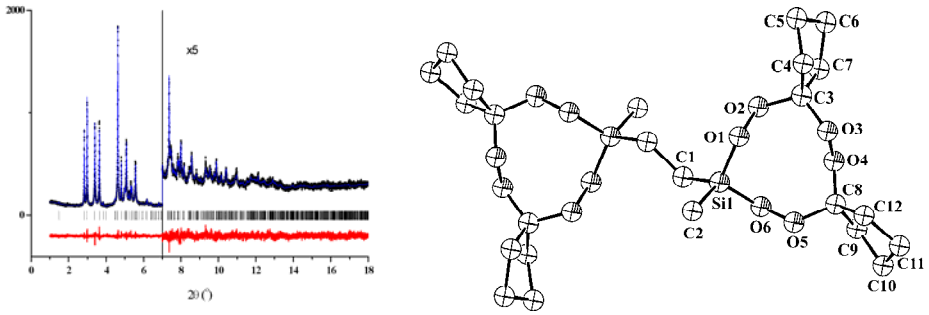
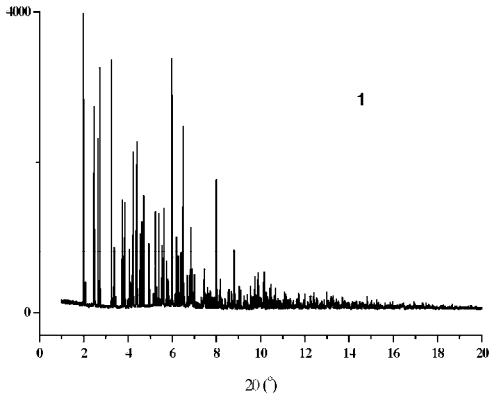



Figure 1. The Rietveld plot and molecular structure for **22**.

Patterns **23** – **30** are in progress.

Finally, we estimate this experimental work as very fruitful and thank the ESRF staff for offering us this wonderful opportunity.

Dr. V.V. Chernyshev
M.V.Lomonosov Moscow State University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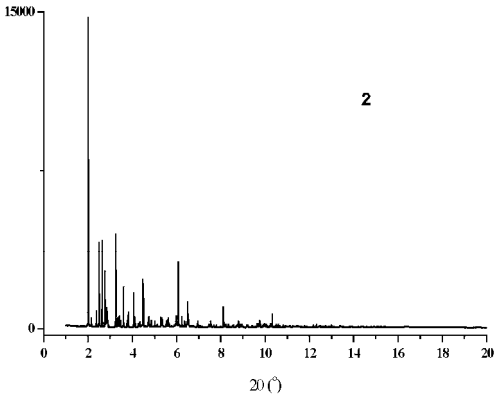
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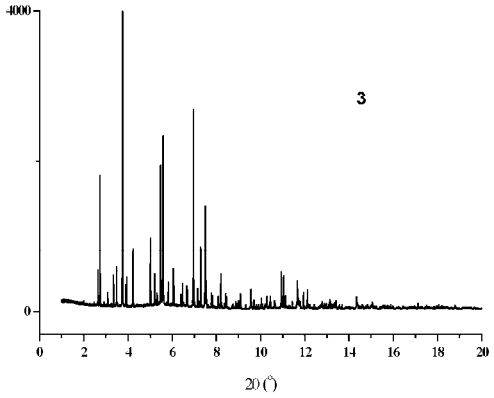
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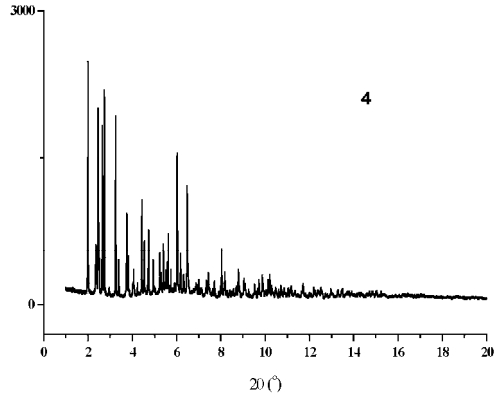
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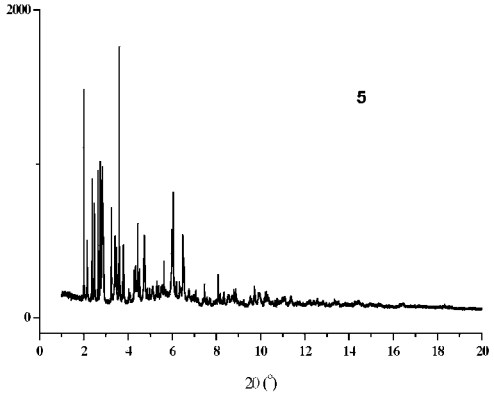
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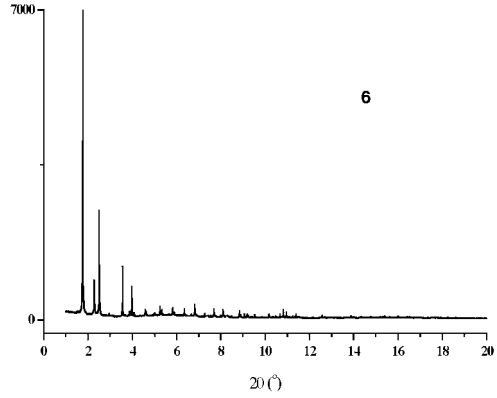
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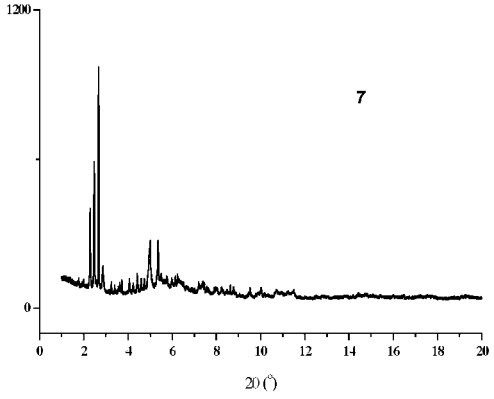
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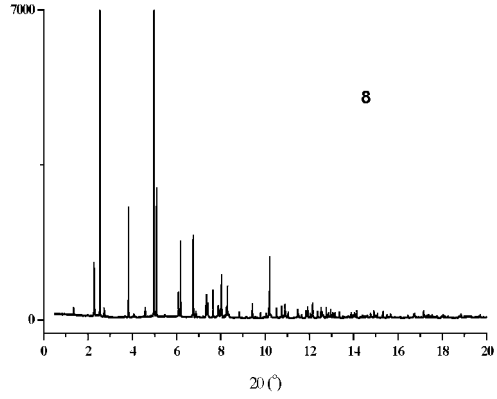
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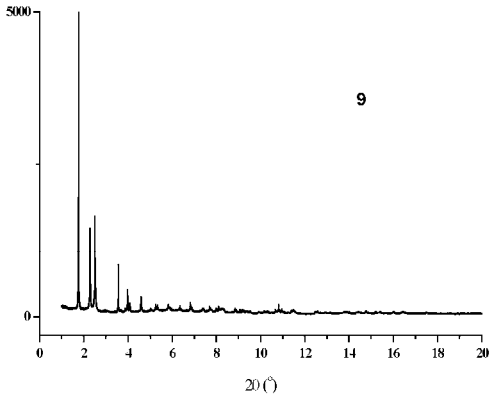
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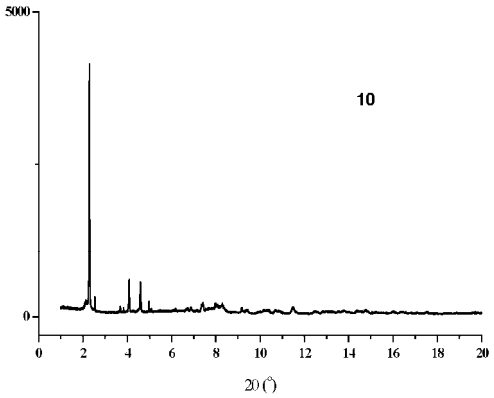
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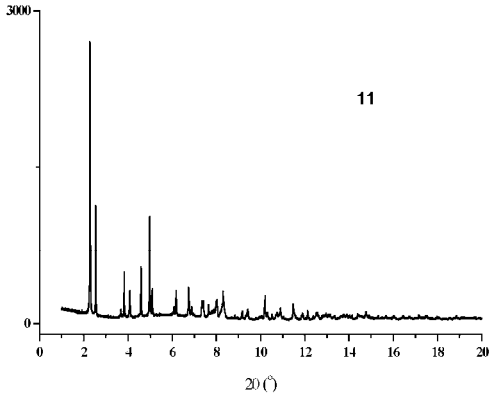
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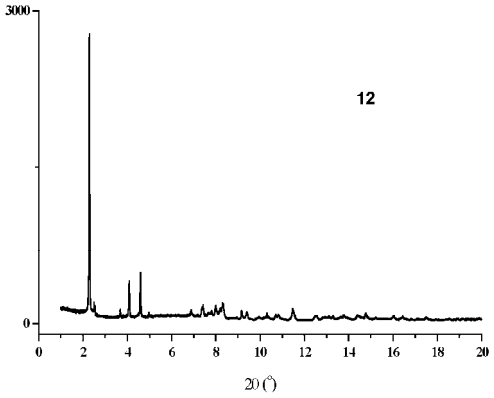
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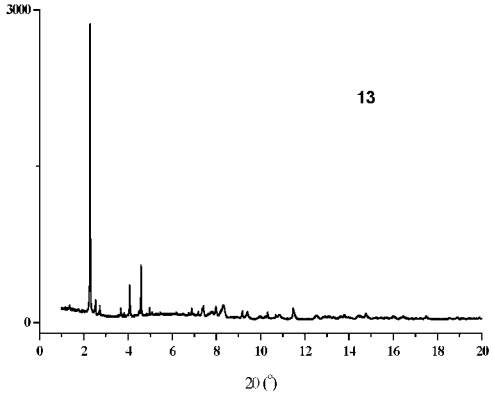
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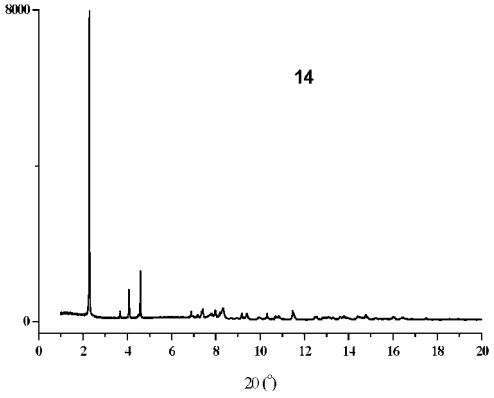
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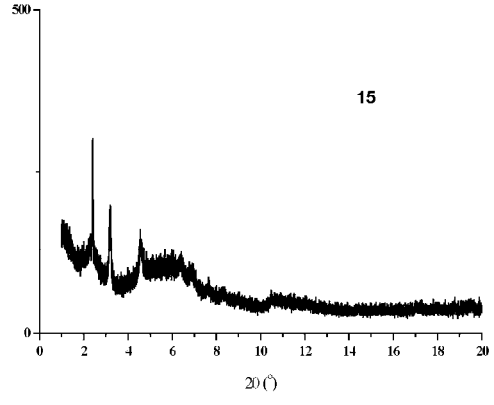
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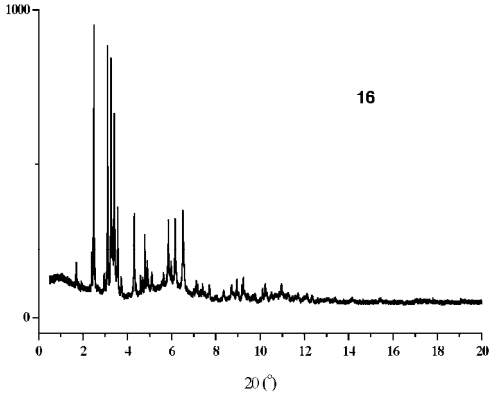
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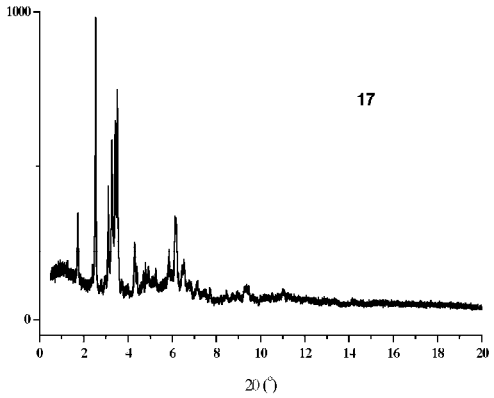
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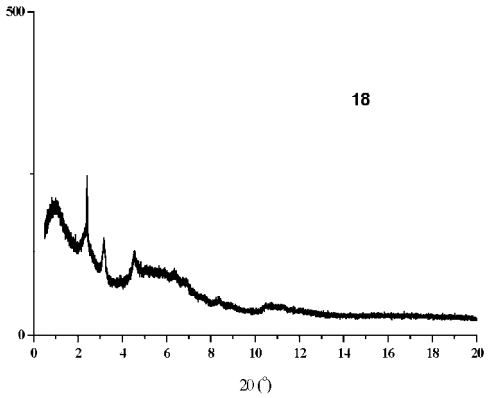
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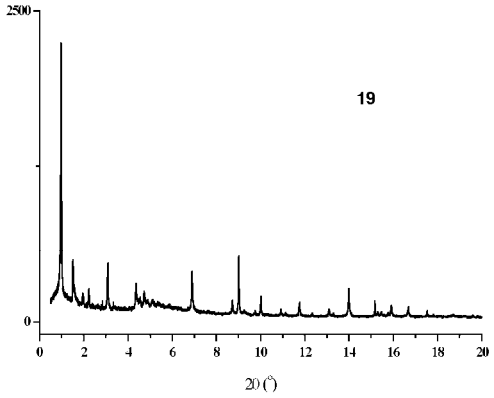
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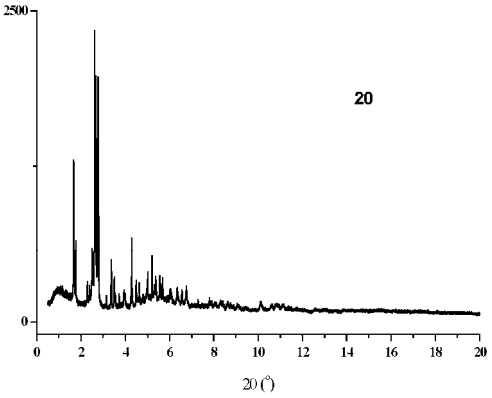
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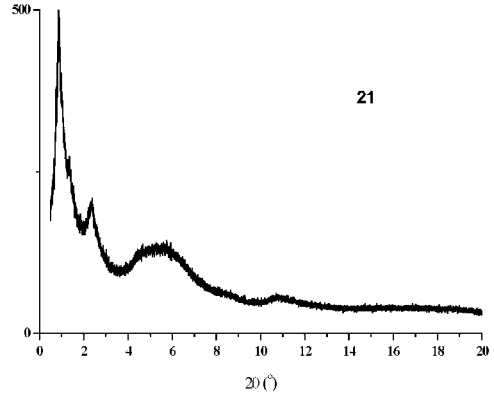
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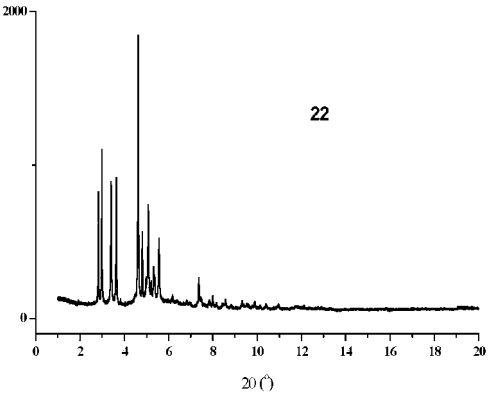
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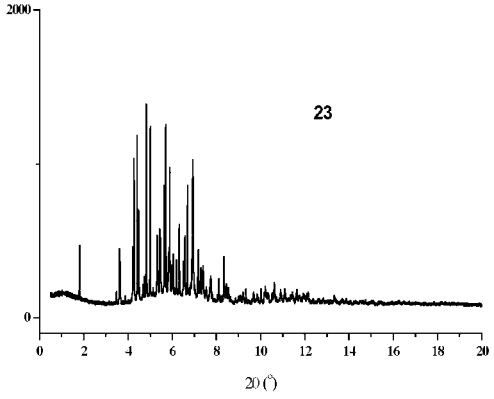
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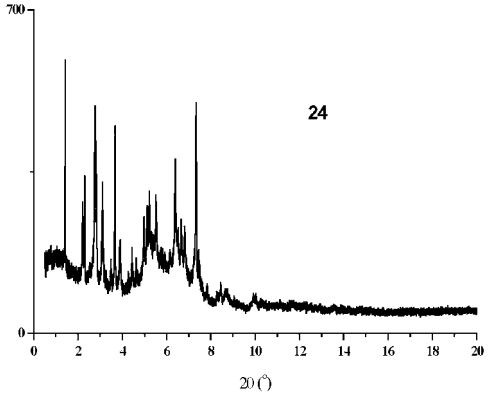
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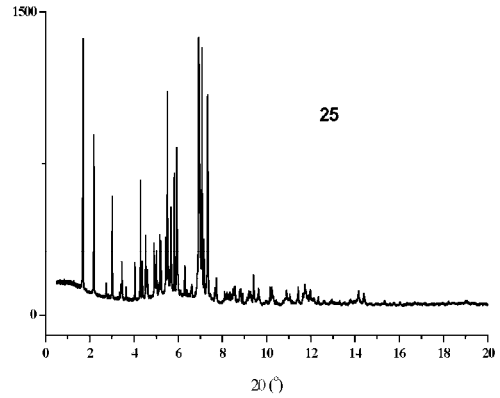
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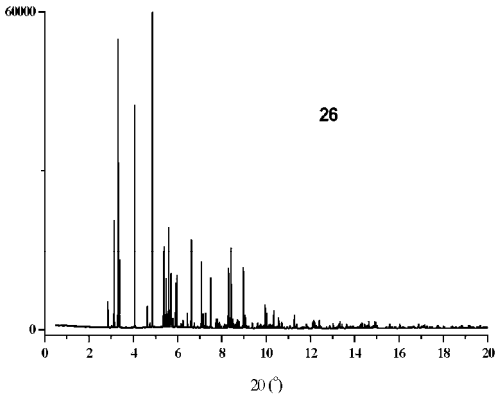
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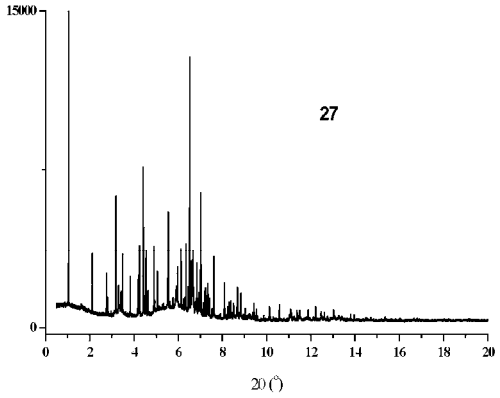
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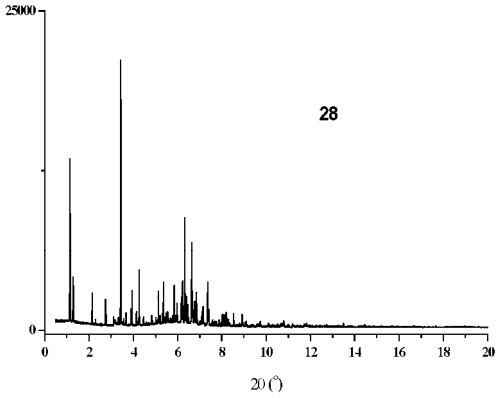
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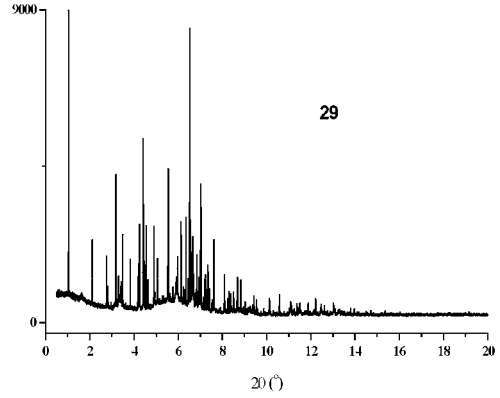
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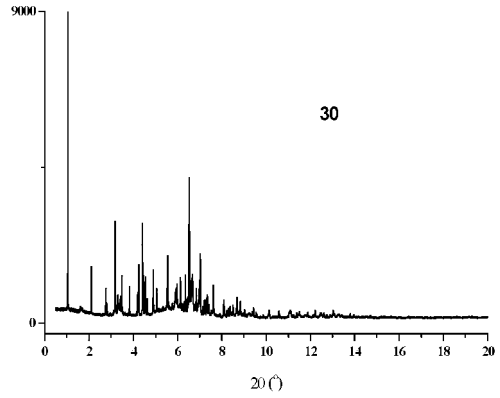
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