The structure determination of germanate zeolites is a challenging work, due to the difficulty in growing sufficiently large crystals suitable for the single crystal X-ray diffraction. Solving the structure by powder X-ray diffraction data combined with electron diffraction is an effective way, though the peak overlap in powder X-ray diffraction patterns, and the instability of germanates under electron beam bring extra task in solving the structure. By combining powder X-ray diffraction, rotation electron diffraction (RED), NMR and IR spectroscopy, a new interrupted zeolite PKU-14 was solved. The $^{19}$F NMR spectrum suggests D4R units exist in the structure. The IR spectrum of PKU-14 shows some similarity with that of ASU-7 within the range 400-1000 cm$^{-1}$, which may reveal PKU-14 could be a zeolitic structure. However, the stretching vibrations of -OH groups at about 3500 cm$^{-1}$ indicates that the structure might be an interrupted zeolite. The three-dimensional reciprocal lattice PKU-14 was reconstructed from the RED data, and from which the unit cell was determined to be $a=19.058$ Å, $b=19.039$ Å, $c=27.365$ Å, $\alpha=89.89^\circ$, $\beta=89.97^\circ$, $\gamma=89.62^\circ$ using the RED software package[1]. From the reflection conditions, it indicates that the possible space group could be $I4cm$, $I-4c2$, $I4/mcm$. Because more than 90% of the zeolite crystal structures in the IZA database are centrosymmetric, most attention was paid to space group $I4/mcm$.

Finally, the structure was solved by using powder X-ray diffraction data with a simulated annealing parallel tempering algorithm using the program FOX[2]. The structure is built by the [4$^6$12$^2$] cages interconnected with D4R units. ITQ-21[3] and ITQ-26[4] are also built by the similar building units, but with a 4MR segment inside the [4$^6$12$^2$] cage. In PKU-14, all the terminal hydroxyl groups point to centre of the [4$^6$12$^2$] cage, but there is no additional species inside it. Therefore, PKU-14 can be considered as a defective structure of ITQ-21. Similar to ITQ-21, PKU-14 also shows a three-dimensional 12MR channel system.

**Keywords:** interrupted zeolite, rotation electron diffraction (RED), structure determination

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