L'originalité de la structure réside dans le fait que les différents feuillets ne sont reliés que par les forces de van der Waals, ce qui explique les difficultés rencontrées lors du choix d'un monocristal. On se trouve donc en présence d'un véritable cristal moléculaire dont la cohésion est assurée par les interactions O(2)—O(3) indiquées sur la Fig. 1(*a*). La distance O(2)—O(3) = 2,91 (1) Å, qui constitue la distance la plus courte entre les feuillets, correspond d'ailleurs bien à une distance de van der Waals pour l'atome d'oxygène (Pauling, 1965).

Bien que de tels cristaux soient plus rares dans les composés minéraux que dans les composés organiques, beaucoup de composés bidimensionnels minéraux ont déja étá décrits et étudiés pour l'anisotropie de leurs propriétés physiques et leur possibilité d'intercaler d'autres molécules. Toutefois, il semble que ce borate d'uranium soit peu propice pour cette dernière propriété puisque les études chimiques antérieures (Hoekstra, 1967) ont prouvé qu'il était insoluble dans l'eau à 298 K et qu'il s'hydrolysait au bout de plusieurs jours en $UO_2(OH)_2$ et en acide borique. Elles ont aussi montré que c'était un des sels d'uranium les plus stables thermiquement et qu'il ne se décomposait qu'au-dessus de 1073 K.

En conclusion, il ressort de l'étude structurale de UB_2O_6 que ce composé est un lamellaire vrai. Sa présence n'est pas à exclure d'éventuels produits de recristallisation susceptibles de se former parmi les verres des déchets nucléaires.

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Structure of Tetratellurium Decachloro-μ-oxo-diniobate

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Abstract. M_r for $Te_4[Nb_2Cl_{10}O] = 1066{\cdot}7$, triclinic, space group $P\bar{1}$, $a = 6{\cdot}507\ (2)$, $b = 8{\cdot}554\ (1)$, $c = 8{\cdot}894\ (2)$ Å, $\alpha = 80{\cdot}35\ (1)$, $\beta = 85{\cdot}22\ (2)$, $\gamma = 73{\cdot}75\ (2)°$, $V = 468{\cdot}2$ Å^3, $Z = 1$, $D_x = 3{\cdot}78$ g cm^{-3}, Mo $K\alpha$ radiation, $\lambda = 0{\cdot}71069$ Å, $\mu = 87{\cdot}3$ cm^{-1}, $F(000) = 468$, $T = 298$ K, $R = 0{\cdot}0378$ for 2321 unique reflections with $I > 3\sigma(I)$. The compound is prepared from the melt of Te, $TeCl_4$, $NbCl_5$ and $NbOCl_3$ in appropriate stoichiometry. The structure consists of a square Te_4^{2+} cation and an $Nb_2OCl_{10}^{2-}$ anion. The anion contains a linear Nb—O—Nb bond which contains eclipsed Cl atoms, and is involved in considerable ion pairing with the Te_4^{2+} cation through asymmetric Cl bridges. These are out of the plane of the Te_4^{2+} ring and result in lengthening of the Te bonds.

Introduction. The Te_4^{2+} ion was first prepared as the $AlCl_4^-$ salt from the reaction of tellurium with tellurium tetrachloride and aluminium trichloride in the molten state (Couch, Lokken & Corbett, 1972).

$$7Te + TeCl_4 + 4AlCl_3 \rightarrow 2Te_4(AlCl_4)_2$$

An alternative more general method is the oxidation of tellurium with oxidizing agents such as AsF_5 and SbF_5 in SO_2 solution (Dean, Gillespie & Ummat, 1974). In the course of study of the reactivity of Te_4^{2+}, we attempted to prepare the new compound $Te_4(NbCl_6)_2$ by a modification of the molten-salt method in which $AlCl_3$ was replaced by $NbCl_5$. However, the crystals obtained were invariably twinned. When an equimolar mixture of $NbCl_5$ and $NbOCl_3$ was used, crystals of $Te_4[Nb_2OCl_{10}]$, which were suitable for X-ray crystallography, were obtained.

Experimental. The title compound was prepared by mixing the reactants in the correct stoichiometry in a

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Table 1. *Positional parameters and their e.s.d.'s*

Anisotropically refined atoms are given in the form of the equivalent isotropic thermal parameter defined as:

$$\tfrac{4}{3}[a^2B(1,1) + b^2B(2,2) + c^2B(3,3) + ab(\cos\gamma)B(1,2) + ac(\cos\beta)B(1,3) + bc(\cos\alpha)B(2,3)]$$

where *a, b* and *c* are reciprocal-lattice constants.

	x	y	z	B(Å^2)
Te(1)	0·07820 (8)	0·19875 (5)	−0·01320 (5)	2·863 (8)
Te(2)	0·18114 (8)	−0·09373 (6)	0·16813 (5)	2·939 (9)
Nb(1)	0·48942 (7)	0·35427 (5)	0·36652 (5)	1·485 (8)
Cl(1)	0·8355 (2)	0·1960 (2)	0·4215 (2)	2·72 (3)
Cl(2)	0·1378 (2)	0·5156 (2)	0·2929 (2)	3·06 (3)
Cl(3)	0·3374 (3)	0·1986 (2)	0·5583 (2)	2·87 (3)
Cl(4)	0·4757 (3)	0·1670 (2)	0·1889 (2)	2·84 (3)
Cl(5)	0·6247 (3)	0·5172 (2)	0·1623 (2)	2·80 (3)
O(1)	0·500	0·500	0·500	2·3 (1)

Table 2. *Selected bond lengths* (Å), *significant contacts* (Å) *and related bond angles* (°)

Te(1)–Te(2)	2·6831 (4)	Nb(1)–Cl(1)	2·324 (1)
Te(1)–Te(2$^{\mathrm{i}}$)	2·6841 (5)	–Cl(2)	2·398 (1)
Te(1)–Cl(2$^{\mathrm{ii}}$)	3·280 (1)	–Cl(3)	2·314 (1)
–Cl(4)	3·196 (1)	–Cl(4)	2·453 (1)
–Cl(5$^{\mathrm{iii}}$)	3·837 (1)	–Cl(5)	2·364 (1)
–Cl(5$^{\mathrm{iv}}$)	3·538 (1)	–O(1)	1·8777 (3)
Te(2)–Cl(1$^{\mathrm{iii}}$)	3·766 (1)	Cl(1)···Cl(1)	3·561 (2)
–Cl(1$^{\mathrm{v}}$)	3·607 (1)	Cl(2)···Cl(2)	3·961 (3)
–Cl(2$^{\mathrm{vi}}$)	3·422 (1)	Cl(3)···Cl(3)	3·727 (3)
–Cl(4)	3·358 (1)	Cl(5)···Cl(5)	3·520 (2)
–Cl(4$^{\mathrm{vii}}$)	3·763 (1)		
–Cl(5$^{\mathrm{vi}}$)	3·751 (1)		

Te(2$^{\mathrm{i}}$)–Te(1)–Te(2)	90·48 (1)	Cl(1)–Nb(1)–Cl(2)	176·32 (5)
Te(1$^{\mathrm{i}}$)–Te(2)–Te(1)	89·52 (1)	–Cl(3)	92·88 (5)
Te(2)–Te(1)–Cl(2$^{\mathrm{ii}}$)	159·62 (3)	–Cl(4)	88·52 (4)
–Cl(4)	69·03 (2)	–Cl(5)	90·28 (5)
–Cl(5$^{\mathrm{iii}}$)	112·42 (2)	–O(1)	91·76 (3)
–Cl(5$^{\mathrm{iv}}$)	132·57 (3)	Cl(2)–Nb(1)–Cl(3)	89·50 (4)
Te(2$^{\mathrm{i}}$)–Te(1)–Cl(2$^{\mathrm{ii}}$)	69·21 (3)	–Cl(4)	88·68 (5)
–Cl(4)	156·69 (3)	–Cl(5)	87·26 (4)
–Cl(5$^{\mathrm{iii}}$)	95·06 (2)	–O(1)	91·00 (4)
–Cl(5$^{\mathrm{iv}}$)	126·49 (2)	Cl(3)–Nb(1)–Cl(4)	89·85 (5)
Te(1)–Te(2)–Cl(1$^{\mathrm{iii}}$)	75·77 (2)	–Cl(5)	176·42 (5)
–Cl(1$^{\mathrm{v}}$)	130·89 (2)	–O(1)	90·93 (4)
–Cl(2$^{\mathrm{vi}}$)	153·10 (3)	Cl(4)–Nb(1)–Cl(5)	88·56 (4)
–Cl(4)	62·72 (2)	–O(1)	179·16 (3)
–Cl(4$^{\mathrm{vii}}$)	75·94 (2)	Cl(5)–Nb(1)–O(1)	90·64 (3)
–Cl(5$^{\mathrm{vi}}$)	134·39 (2)	Nb(1)–O(1)–Nb(1)	180·0
Te(1$^{\mathrm{i}}$)–Te(2)–Cl(1$^{\mathrm{iii}}$)	107·93 (2)	Nb(1$^{\mathrm{ii}}$)–Cl(2$^{\mathrm{ii}}$)–Te(1)	137·71 (5)
–Cl(1$^{\mathrm{v}}$)	117·48 (2)	Nb(1$^{\mathrm{ii}}$)–Cl(2$^{\mathrm{ii}}$)–Te(2$^{\mathrm{i}}$)	105·81 (4)
–Cl(2$^{\mathrm{vi}}$)	63·63 (2)	Nb(1)–Cl(4)–Te(1)	125·49 (5)
–Cl(4)	150·16 (2)	Nb(1)–Cl(4)–Te(2)	135·18 (5)
–Cl(4$^{\mathrm{vii}}$)	80·20 (2)		
–Cl(5$^{\mathrm{vi}}$)	92·91 (2)		
Te(2$^{\mathrm{i}}$)–Cl(2)–Te(1)	47·16 (2)		
Te(1)–Cl(4)–Te(2)	48·25 (2)		

Symmetry operations: (i) $-x, -y, -z$; (ii) $-x, 1-y, -z$; (iii) $-1 + x, y, z$; (iv) $1-x, 1-y, -z$; (v) $1-x, -y, 1-z$; (vi) $x, -1+y, z$; (vii) $1-x, -y, -z$.

Pyrex tube under dry N_2. The tube was evacuated, sealed and heated at 475 K for 1 week, after which time black crystals were transported from the melt to the top of the tube.

$$7\mathrm{Te} + \mathrm{TeCl_4} + 2\mathrm{NbCl_5} + 2\mathrm{NbOCl_3} \rightarrow 2\mathrm{Te_4[Nb_2OCl_{10}]}$$

Black plates and blocks of $\mathrm{Te_4[Nb_2OCl_{10}]}$ were selected and sealed in Lindemann capillaries under N_2. Precession photographs were taken to determine crystal quality and to obtain preliminary space-group information. Further work was performed on an Enraf–Nonius CAD-4 diffractometer using graphite-monochromated Mo $K\alpha$ radiation. Cell dimensions obtained by least-squares refinement of 25 reflections, range $9{\cdot}5 < \theta < 16{\cdot}0°$; intensity data using θ:2θ scans over scan ranges $(0{\cdot}80 + 0{\cdot}35 \tan\theta)°$; variable scan rates, $I/\sigma(I)$ ratio of 25 within maximum scan time 80 s; prescans at 10·1° min^{-1}. For each reflection, backgrounds obtained extending the scan 25% on either side of the peak; 3003 reflections in quadrants $(0 \le h \le 9, -11 \le k \le 11, -12 \le l \le 12)$ with $2\theta < 60°$ measured. Repeat measurement of three standard reflections every 8200 s of exposure time showed no systematic variations. Lorentz and polarization corrections applied to all reflections; absorption corrections applied using program *ABSORB* and 10 × 8 × 8 grid; crystal faces identified as (011), (011), (011), (011) and (100) at 0·027, 0·027, 0·069, 0·069 and 0·087 mm respectively from origin within crystal. Transmission coefficients from 0·331 (011) to 0·637 (111); 2642 reflections obtained after averaging 402 symmetry-equivalent data ($R_{\mathrm{merge}} = 0{\cdot}01$) excluding data with $I \le 0{\cdot}0$. Structure solved using Patterson synthesis to locate heavy atoms; all other atoms located after several cycles of least-squares and Fourier calculations. Full-matrix least-squares refinement minimizing $\sum w\Delta F^2$ and including extinction corrections converged [maximum shift/e.s.d. 0·23 (for g)] to final agreement indices $R = 0{\cdot}0378$ ($wR = 0{\cdot}0577$) for 2321 reflections with $I > 3\sigma(I)$; final cycle weights given by $\omega = 4F^2[\sigma(I)^2 + (0{\cdot}04F^2)^2]^{-1}$. At convergence, e.s.d. of observation of unit weight was 1·95; largest peak in final difference Fourier map less than 1·5 e $\mathrm{Å}^{-3}$; final value of g 4·8 (6) $\times 10^{-7}$. PDP 11/23 computer and programs in the Enraf–Nonius *SDP* package used for the refinements (B. A. Frenz & Associates, Inc., 1981); atom scattering factors from *International Tables for X-ray Crystallography* (1974); final positional parameters and selected bond lengths and bond angles are given in Tables 1 and 2.

Discussion. The compound contains $\mathrm{Te_4^{2+}}$ cations and $\mathrm{Nb_2OCl_{10}^{2-}}$ anions with appreciable interactions.* The $\mathrm{Te_4^{2+}}$ cation has crystallographic inversion symmetry with Te—Te bond lengths 2·6831 (4) and 2·6841 (5) Å and is very slightly distorted from square-planar D_{4h} symmetry. The Te—Te bond lengths are slightly longer than in the $\mathrm{Te_4^{2+}}$ cation in $\mathrm{Te_4(AlCl_4)_2}$ [2·662 (2) and 2·627 (2) Å], $\mathrm{Te_4(Al_2Cl_7)_2}$ [2·660 (2) and 2·661 (2) Å]

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* Lists of general temperature factors and final structure-factor amplitudes (calculated and observed) have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44112 (17 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

(Couch, Lokken & Corbett, 1972) and $Te_4(SbF_6)_2$ [2·669 (3) and 2·676 (3) Å] (Cardinal, Gillespie, Sawyer & Vekris, 1982). The longer Te—Te bonds may be caused by the appreciably stronger anion–cation interactions in the present compound, although we have no explicit bond-length–bond-strength model. Similar interactions are a characteristic feature of the crystal packing of all M_4^{2+} cations (M = S, Se and Te) and have been discussed elsewhere (Cardinal, Gillespie, Sawyer & Verkris, 1982). In the title compound the edges of the cation are asymmetrically bridged by atoms Cl(2) and Cl(4) which are 0·095 (2) and 0·589 (2) Å respectively out of the plane of the cation (Fig. 1). The bridging Te···Cl distances [3·196 (1)–3·442 (1) Å] are shorter than the analogous distances in $Te_4(AlCl_4)_2$ [3·292 (4)–3·434 (5) Å] and $Te_4(Al_2Cl_7)_2$ [3·290 (5)–3·525 (5) Å]. There are also short Te···Cl contacts along the extensions of the diagonals of the Te_4^{2+} cation in $Te_4[Nb_2OCl_{10}]$ which are approximately 0·10–0·15 Å shorter than comparable interactions in $Te_4(AlCl_4)_2$ and $Te_4(Al_2Cl_7)_2$ [see Table 4 in Cardinal, Gillespie, Sawyer & Vekris (1982)].

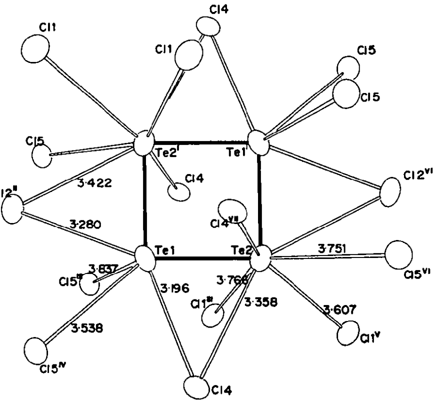

Fig. 1. *ORTEP* (Johnson, 1965) view of the Te_4^{2+} molecule showing the short Te···Cl contacts.

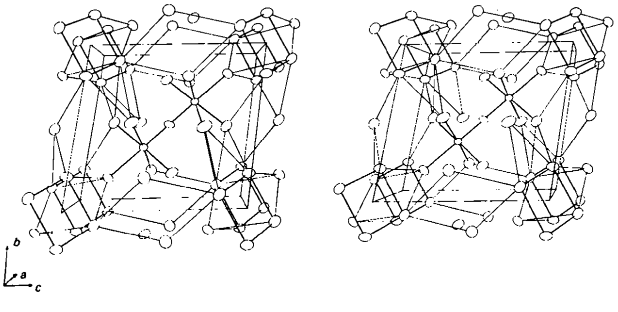

Fig. 2. Stereoview of the unit cell of $Te_4[Nb_2OCl_{10}]$ showing the orientation of the $Nb_2OCl_{10}^{2-}$ anion with regard to the Te ring.

In the $Nb_2OCl_{10}^{2-}$ anion, the O atom is on a center of symmetry so that the equatorial Cl atoms are eclipsed and the Nb—O—Nb bridge is linear with an Nb—O distance of 1·877 (3) Å (Fig. 2). The Nb—O distance is intermediate between the accepted single- and double-bond lengths of 2·10–2·15 and *ca* 1·70 Å respectively, and is very nearly the same as the bridging Nb—O bond lengths in compounds like $[\{(C_5H_5)_2NbCl\}_2O](BF_4)_2$ [1·88 (1) Å] (Prout, Cameron, Forder, Critchley, Denton & Rees, 1974) and $[\{(C_5H_4CH_3)NbCl_3(H_2O)\}_2O]$ [1·902 (2) Å] (Prout & Daran, 1979). To our knowledge, this anion has not been previously reported, although the analogous $Ta_2OCl_{10}^{2-}$ has been described (Cotton & Najjar, 1981). The tantalum compound is also linear and has an M—O distance that is almost identical to that in the present compound.

The Nb—Cl bond *trans* to the bridging O atom has a length of 2·453 (1) Å, which is significantly longer than the other four Nb—Cl bonds [2·324 (1)–2·398 (1) Å]. This *trans* effect can partly be attributed to the bridging O atom as discussed by Cotton & Najjar (1981). However, the effect observed here is significantly larger than that observed in the Ta compound in which no ion-pairing effects were present (0·045 *versus* 0·103 Å in the present compound). Thus, part of this observed effect is probably due to the bridging interaction of the *trans* Cl with the Te_4^{2+} cation. The significant differences in the lengths of the equatorial Nb—Cl bonds also correlate well with the lengths of the intermolecular contacts (Table 2). Some of these Nb—Cl bonds are significantly longer than the bonds in the $NbCl_6^-$ ion [2·20–2·34 (2) Å in $PCl_4^+.NbCl_6^-$ (Preiss, 1971)]. They are, however, significantly shorter than the analogous Nb—Cl bond lengths in $[\{(C_5H_4CH_3)NbCl_3(H_2O)\}_2O]$ (2·449 Å average).

In the $Nb_2OCl_{10}^{2-}$ anion, the four equatorial Cl atoms bend by *ca* 1° towards the longest Nb—Cl(4) bond. Other small differences between bond angles also correlate well with observed bond lengths.

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$CsMoP_2O_7$: a Molybdenopyrophosphate Containing Isolated Mo^{3+} Cations

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Abstract. $CsMoP_2O_7$, $M_r = 402{\cdot}793$, monoclinic, $P2_1/c$, $a = 7{\cdot}7239\,(22)$, $b = 10{\cdot}304\,(10)$, $c = 8{\cdot}4881\,(15)$ Å, $\beta = 104{\cdot}772\,(17)^\circ$, $V = 653\,(1)$ Å^3, $Z = 4$, $D_x = 4{\cdot}10$ g cm^{-3}, $\lambda(\text{Mo } K\alpha) = 0{\cdot}71069$ Å, $\mu = 80{\cdot}22$ cm^{-1}, $F(000) = 732$, $T = 296$ K, $R = 0{\cdot}041$ for 915 independent reflections. The title compound is isostructural with $KAlP_2O_7$ and contains unusual isolated Mo^{3+} cations in the oxygen array. The pyrophosphate groups form sheets linked by Mo atoms. The MoO_6 octahedra are slightly distorted. The Cs ions are located in tunnels formed by the polyhedra and are coordinated by ten O atoms.

Introduction. The investigations of the system M–Mo^{V}–P–O (M = metal cation) have shown the formation of $K_4Mo_8P_{12}O_{52}$ (Leclaire, Monier & Raveau, 1983), $Cs_2Mo_4P_6O_{26}$ (Lii & Haushalter, 1987), $Cs_4Mo_8P_{12}O_{52}$ (Lii & Haushalter, 1987) and $AgMo_5P_8O_{33}$ (Lii, Johnston, Goshorn & Haushalter, 1987). A phosphate containing Mo^{IV} has been observed in $TlMo_2P_3O_{12}$ (Leclaire, Monier & Raveau, 1985). In contrast to the Mo^{V}-containing compounds, which exhibit isolated MoO_6 octahedra linked only to PO_4 tetrahedra, $TlMo_2P_3O_{12}$ contains corner-sharing Mo_2O_{11} units and PO_4 groups. As the formal oxidation state of the Mo atom is further reduced, one might expect the formation of metal–metal bonds which were found in the cubane-like Mo_4O_4 cluster unit in the compounds $Cs_3Mo_5P_6O_{25}$ (Lii, Haushalter & O'Connor, 1987) and $Cs_3Mo_4P_3O_{16}$ (Haushalter, 1987). The interconnection between the building units is governed by several factors such as the nature of the counter cation, the oxidation state of the Mo atom, and the nonmetal-to-metal ratio. Therefore, one might be able to prepare a caesium molybdenophosphate containing isolated highly reduced Mo cations by adjusting the nonmetal-to-metal ratio. Hence, we prepared $CsMoP_2O_7$ which contains unusual isolated Mo^{3+} cations in the oxygen array. This compound was found to be isostructural with $KAlP_2O_7$ (Ng & Calvo, 1973). To convert the $KAlP_2O_7$ coordinates to those of $CsMoP_2O_7$, one must shift the origin in $KAlP_2O_7$ by $-\frac{1}{2}$ in y, then apply the values $1-x$, $y-\frac{1}{2}$, $1{\cdot}5-z$ to all coordinates.

Experimental. Yellow needle crystals of $CsMoP_2O_7$ were discovered in the reaction products formed by heating a mixture of Cs_2MoO_4, Mo and P_2O_5 (mole ratio of 1:1:2) at 1270 K in an evacuated quartz ampule for 32 h. Although reactions to prepare a pure phase at several different temperatures have been performed, the X-ray powder patterns of the products always showed either a few unindexed reflections or reflections due to $Cs_3Mo_5P_6O_{25}$.

The intensity data were collected using an Enraf–Nonius CAD-4 diffractometer and graphite-monochromated Mo $K\alpha$ radiation. 25 independent reflections with $10{\cdot}6 < 2\theta < 50^\circ$ were used for least-squares determination of cell constants. The intensities of three reflections were monitored and no significant decay was detected. The structure was solved by direct methods and refined by full-matrix least-squares refinement based on F values. The Cs, Mo and P atoms were refined anisotropically. Attempts to refine the O atoms anisotropically resulted in negative temperature factors for some of these atoms so they were refined isotropically. A refinement of the occupancy factors for the Cs and Mo atoms resulted in a value of 0·97 (1) for both atoms, indicating full occupancy. All calculations were performed on a VAX-based TEXRAY system. Scattering factors and anomalous-dispersion terms were taken from *International Tables for X-ray Crystallography* (1974). Data-collection and refinement parameters are collected in Table 1.† Final

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† Lists of structure factors, anisotropic thermal parameters and a full list of intramolecular distances have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44194 (10 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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