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Equiatomic Compounds of the Transition and Lanthanide Elements with Rh, Ir, Ni and Pt

BY A. E. DWIGHT, R. A. CONNER, JR. AND J. W. DOWNEY

Argonne National Laboratory, Argonne, Illinois, U.S.A.

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Crystal structures and lattice parameters are reported for a number of new intermetallic compounds. Representatives have been found of the orthorhombic $B19$, B_f and $B27$ and the cubic $B2$ structures.

Introduction

A continuing study is under way on the alloy chemistry of the transition elements. In the course of this study a number of new intermetallic compounds have been discovered. It is the purpose of the present paper to report crystal structure data on equiatomic compounds between an element of the Sc (including the lanthanides), Ti, V or Cr group and an element of the Co or Ni group of the Periodic Table.

Experimental

Alloys were made by arc melting on a water-cooled copper hearth under an argon atmosphere. The weight loss during melting varied, but even for the most difficult alloy, TmIr, it was less than 1%. Crystal bar titanium, zirconium and hafnium were used. Rare earth elements were 99·9% pure, as also were the nickel, rhodium, platinum and iridium. The arc-melted buttons were homogenized in evacuated capsules at temperatures appropriately below their melting points. Powder specimens for X-ray diffraction were then prepared and heat treated briefly at the same temperature of homogenization. For the rhodium alloys only, a second set of powder specimens was slowly cooled from 905 °C to 600 °C, held at 600 ° for 24 hours, then slowly cooled to room temperature. Metallographic specimens were examined for evidence of segregation or liquation.

X-ray powder photographs were taken with Cr $K\alpha$ or Cu $K\alpha$ radiation and a Straumanis-type Debye–Scherrer powder camera (114·6 mm diameter). For patterns showing a b.c. cubic structure, the lattice parameters were obtained by the Nelson–Riley extrapolation method. For patterns showing an orthorhombic structure, indices were obtained by use of the Battelle Indexing Charts (Bell & Austin, no date). Lattice parameters were obtained from the IBM 704 computer by the method described by Mueller & Heaton (1961). The computer was also used to calculate d spacings (for comparison with observed d spacings) and to calculate F^2. Intensity calculations were made, by means of the relation $I \sim (F^2)$ (Lp factor) (multiplicity), to verify the assignment of indices.

Results and discussion

The $B19$ structure was found in the equiatomic TiPd and TiPt compounds. TiPt was isolated but not identified by Nishimura & Hiramatsu (1957). Their work showed the compound to be formed by an open maximum transformation at 1830 °C. The $B19$ structure reported by Schubert, Frank, Gohle, Maldonado, Meissner, Rama & Rossteutscher (1963) in VPt and MoPt is confirmed, as is also the $B19$ structure in NbPt (Giessen & Grant, 1964). Lattice parameters and probable errors of these compounds are given in Table 1; d spacings and intensities of TiPt are given in Table 2.

In the $B19$ structure the atomic positional parameters x and y are fixed at $\frac{1}{4}$ and $\frac{1}{2}$ for Ti and $\frac{1}{4}$ and 0 for Pt. The z positional parameters vary, although the difference between the two z parameters is fixed at 0·50 by the geometry of the unit cell (Steeple, 1952). Intensities for TiPt were calculated with three sets of z positional parameters: 0·16 and 0·66, 0·18 and 0·68, 0·20 and 0·70. The best agreement with observed intensities was obtained with $z=0{\cdot}18$ for Ti and $z=0{\cdot}68$ for Pt, values which are close to those reported by Steeple (1952) for the $B19$ structure in MgCd: $z=0{\cdot}177$ for Mg and $z=0{\cdot}682$ for Cd. Only two other examples of the $B19$ structure have been reported, *i.e.* AuCd and TiAu (Pearson, 1958).

The B_f (CrB-type) structure was found in fourteen compounds, which are listed in Table 1 with their lattice parameters. We confirm the existence of a B_f structure in CeNi, as reported by Tinney & Rosenzweig (1961). Our results for GdNi disagree with those of earlier workers. Novy, Vickery & Kleber (1961) reported GdNi to be orthorhombic, from single crystal data, with $a_o=3{\cdot}8$, $b_o=5{\cdot}2$ and $c_o=4{\cdot}2$ Å. If the b_o value were doubled the lattice parameters by Novy *et al.* would be in fair agreement with those obtained in the present investigation. In contrast, Baenziger & Moriarty (1961) reported GdNi to have a $B27$ (FeB-type) structure, with $a_o=5{\cdot}428$, $b_o=4{\cdot}353$ and $c_o=6{\cdot}931$ Å. To support the B_f structure which we find for GdNi, calculated and observed d spacings and intensities are given in Table 3. In the B_f structure, the atomic positional parameters x and z are respectively 0 and 0·25 for both Gd and Ni. Intensity cal-

Table 1. *Lattice parameters of equiatomic compounds*

AB	Type	a_o	b_o	c_o	Metallographic observations
TiPt	*B*19	4·592 ± 0·001	2·761 ± 0·001	4·838 ± 0·001	single phase
TiPd	*B*19	4·56 ± 0·01	2·81 ± 0·01	4·89 ± 0·01	1 % second phase
VPt	*B*19	4·413 ± 0·001	2·692 ± 0·001	4·770 ± 0·001	single phase
VPt(1)	*B*19	4·42	2·69	4·76	
NbPt	*B*19	4·62 ± 0·01	2·78 ± 0·01	4·98 ± 0·01	2 % second phase
NbPt(2)	*B*19	4·611	2·780	4·983	
MoPt	*B*19	4·475 ± 0·001	2·729 ± 0·001	4·914 ± 0·001	single phase
MoPt(1)	*B*19	4·47	2·74	4·89	
ZrPt	B_f	3·409 ± 0·001	10·315 ± 0·001	4·277 ± 0·001	6 % second phase
HfPt	B_f	3·345 ± 0·001	10·269 ± 0·001	4·288 ± 0·001	1 % second phase
LaNi	B_f	3·907 ± 0·001	10·810 ± 0·001	4·396 ± 0·001	2 % second phase
CeNi	B_f	3·788 ± 0·001	10·556 ± 0·001	4·366 ± 0·001	2 % second phase
CeNi(3)	B_f	3·77 ± 0·01	10·46 ± 0·02	4·37 ± 0·01	
PrNi	B_f	3·816 ± 0·001	10·503 ± 0·001	4·354 ± 0·001	2 % second phase
NdNi	B_f	3·803 ± 0·001	10·461 ± 0·001	4·339 ± 0·001	2 % second phase
SmNi	B_f	3·776 ± 0·001	10·358 ± 0·001	4·291 ± 0·001	1 % second phase
GdNi	B_f	3·766 ± 0·001	10·316 ± 0·001	4·244 ± 0·001	1 % second phase
LaRh	B_f	3·986 ± 0·001	11·144 ± 0·001	4·245 ± 0·001	1 % second phase
CeRh	B_f	3·852 ± 0·001	10·986 ± 0·001	4·152 ± 0·001	5 % second phase
PrRh	B_f	3·905 ± 0·001	10·910 ± 0·001	4·210 ± 0·001	5 % second phase
NdRh	B_f	3·890 ± 0·002	10·839 ± 0·007	4·247 ± 0·004	5 % second phase
LaPt	B_f	3·974 ± 0·001	11·037 ± 0·001	4·558 ± 0·001	single phase
CePt	B_f	3·921 ± 0·001	10·920 ± 0·001	4·524 ± 0·001	single phase
DyNi	*B*27	7·043 ± 0·001	4·164 ± 0·001	5·451 ± 0·001	1 % second phase
DyNi(4)	*B*27	6·895 ± 0·002	4·319 ± 0·002	5·353 ± 0·004	
HoNi	*B*27	7·022 ± 0·001	4·140 ± 0·001	5·435 ± 0·001	single phase
ErNi	*B*27	6·991 ± 0·001	4·114 ± 0·001	5·418 ± 0·001	1 % second phase
TmNi	*B*27	6·959 ± 0·001	4·099 ± 0·001	5·398 ± 0·001	1 % second phase
LuNi	*B*27	6·912 ± 0·001	4·073 ± 0·001	5·366 ± 0·001	10 % second phase
YNi	*B*27	7·151 ± 0·001	4·124 ± 0·001	5·513 ± 0·001	1 % second phase
YNi(5)	*B*27	7·12 ± 0·02	4·10 ± 0·02	5·51 ± 0·02	
YPt	*B*27	7·010 ± 0·001	4·471 ± 0·001	5·552 ± 0·001	15 % second phase
PrPt	*B*27	7·294 ± 0·001	4·560 ± 0·001	5·698 ± 0·001	15 % second phase
NdPt	*B*27	7·256 ± 0·001	4·551 ± 0·001	5·675 ± 0·001	10 % second phase
SmPt	*B*27	7·152 ± 0·001	4·525 ± 0·001	5·626 ± 0·001	single phase
GdPt	*B*27	7·088 ± 0·001	4·502 ± 0·001	5·590 ± 0·001	5 % second phase
GdPt(4)	*B*27	7·164 ± 0·003	4·458 ± 0·003	5·574 ± 0·005	
TbPt	*B*27	7·018 ± 0·001	4·494 ± 0·001	5·561 ± 0·001	5 % second phase
DyPt	*B*27	6·983 ± 0·001	4·478 ± 0·001	5·544 ± 0·001	15 % second phase
DyPt(4)	*B*27	7·118 ± 0·009	4·453 ± 0·013	5·466 ± 0·001	
HoPt	*B*27	6·951 ± 0·001	4·470 ± 0·001	5·532 ± 0·001	5 % second phase
ErPt	*B*27	6·904 ± 0·001	4·453 ± 0·001	5·512 ± 0·001	5 % second phase
TmPt	*B*27	6·855 ± 0·001	4·446 ± 0·001	5·496 ± 0·001	20 % second phase
LuPt	*B*27	6·810 ± 0·001	4·417 ± 0·001	5·479 ± 0·001	5 % second phase
ScRh	*B*2	3·204 ± 0·001			5 % second phase
ScRh(6)	*B*2	3·206 ± 0·001			
*YRh	*B*2	3·407 ± 0·002			5 % second phase
*SmRh	*B*2	3·464 ± 0·002			5 % second phase
*GdRh	*B*2	3·440 ± 0·002			3 phase
*TbRh	*B*2	3·417 ± 0·001			1 % second phase
*DyRh	*B*2	3·403 ± 0·002			single phase
*HoRh	*B*2	3·388 ± 0·002			2 % second phase
*ErRh	*B*2	3·372 ± 0·002			1 % second phase
TmRh	*B*2	3·358 ± 0·002			2 % second phase
LuRh	*B*2	3·332 ± 0·002			single phase
*HoIr	*B*2	3·383 ± 0·002			20 % second phase
*ErIr	*B*2	3·367 ± 0·002			40 % second phase
*TmIr	*B*2	3·353 ± 0·002			10 % second phase
*LuIr	*B*2	3·332 ± 0·002			10 % second phase

(1) Schubert *et. al.* (1963).
(2) Giessen & Grant (1963).
(3) Tinney & Rosenzweig (1961).
(4) Baenziger & Moriarty (1961).
(5) Beaudry & Daane (1960).
(6) Compton (1958).

* Superlattice lines not detected.

Table 2. *Calculated and observed d spacings and intensities for* TiPt

Wavelength of radiation: Cr $K\alpha_1 = 2{\cdot}28962$ Å

hkl	d_c	d_o	I_c	I_o
001	4.838		182	*vvw*
101	3·329	3·31	655	*ms*
010	2·76	2·75	241	*ms*
002	2·42	2·41	188	*ms*
011	2·40	2·40	165	*w*
200	2·294	2·29	395	*s*
102	2·14	2·135	380	*ms*
111	2·122	2·12	1000	*vs*
201	2·073	2·07	38	*w*
012	1·819	1·82	58	*w*
210	1·765	1·763	132	*m*
202	1·665	1·665	128	*ms*
211	1·658	1·66	114	*m*
003	1·613	1·61	53	*m*
103	1·521	—	6	—
301	1·459	1·459	83	*m*
212	1·425	1·425	84	*m*
013	1·392	1·392	253	*ms*
020	1·381	1·380	138	*ms*
113	1·332	1·330	36	*w*
021	1·327	—	21	—
203	1·320	1·320	110	*ms*
302	1·293	1·293	190	*ms*
311	1·290	1·290	523	*vs*
121	1·275	1·275	215	*s*
004	1·210	—	8	—
022	1·199	1·199	292	*m*
213	1·190	1·19	1017	*vs*
220	1·183	1·183	562	*vs*
312	1·171	1·170	293	*vs*
104	1·170		642	
122	1·160	1·16	903	*ms*

culations were made with $y = 0{\cdot}135$ for Gd and $y = 0{\cdot}425$ for Ni. Agreement with observed intensities was only fair. A second set of calculations with $y = 0{\cdot}135$ for Gd and $y = 0{\cdot}435$ for Ni gave much better agreement. The calculated intensities in Table 3 are based on the latter set of positional parameters.

Table 3. *Calculated and observed d spacings and intensities for* GdNi

Wavelength of radiation: Cr $K\alpha_1 = 2{\cdot}28962$ Å

hkl	d_c	d_o	I_c	I_o
020	5·158	—	44	—
110	3·537	3·523	105	*vw*
021	3·277	3·273	515	*ms*
111	2·717	2·712	1000	*vs*
040	2·579	2·574	265	*m*
130	2·539	2·535	471	*ms*
041	2·204	2·201	137	*m*
131	2·179	2·178	549	*s*
002	2·122	2·118	283	*ms*
022	1·962	—	4	—
200	1·883	1·882	194	*w*
112	1·820	1·82	36	*vw*
150	1·809	—	15	—
220	1·769	—	3	—
060	1·719	—	< 1	—
151	1·664	1·665	89	*w*
042	1·639	1·639	138	*m*
221	1·633	1·629	142	*ms*
132	1·628		255	
061	1·594	1·593	196	*m*
240	1·521	1·520	131	*w*
241	1·432	1·431	98	*w*
202	1·408	1·408	239	*ms*
152	1·377	—	20	—
170	1·372	1·372	215	*ms*
023	1·364	1·365	67	*vw*
222	1·359	—	5	—
062	1·336	1·336	1	*vw*
113	1·313	1·313	229	*ms*
171	1·306	—	55	—
080	1·290	—	29	—
260	1·270	—	1	—
310	1·246	—	17	—
043	1·240	1·241	69	*w*
242	1·236	1·236	359	*s*
133	1·236		305	
081	1·234	—	54	—
261	1·216	1·216	189	*m*
311	1·196	1·196	378	*w*
330	1·179	1·179	247	*w*
172	1·152	1·152	1756	*s*

The equiatomic compound ZrPt was reported by Kendall, Hays & Swift (1961) to be formed at 2100 °C by an open maximum reaction. Kendall *et al.* did not identify the crystal structure of ZrPt, but did present a diffractometer tracing which appears to show the same *d* spacings and intensities that we observe.

The *B*27 (FeB-type) structure was found in many compounds of nickel and platinum, but not of rhodium. Our data for seventeen *B*27 compounds are given in Table 1. YNi was reported earlier by Beaudry & Daane (1960) to be orthorhombic, *mmm* Laue symmetry, with the lattice parameters given in Table 1. Our values for YNi agree closely, although we have chosen different crystallographic axes.

GdPt, DyPt and DyNi were reported as *B*27 structures by Baenziger & Moriarty (1961). We confirm the crystal structure given by Baenziger & Moriarty. Their lattice parameters as well as ours are given in Table 1. To support our determination of the *B*27 structure, intensities and *d* spacings for LuNi are given in Table 4.

Table 4. *Calculated and observed d spacings and intensities for* LuNi

Wavelength of radiation: Cr $K\alpha_1 = 2{\cdot}28962$ Å

hkl	d_c	d_o	I_c	I_o
101	4·239	4·236	5	*vw*
200	3·456	3·452	156	*w*
011	3·244	3·242	474	*ms*
111	2·937	2·935	770	*s*
201	2·905	2·902	406	*ms*
002	2·683	—	77	—
210	2·635	2·636	901	*s*
102	2·501	2·501	894	*s*
211	2·365	2·367	1000	*vs*
112	2·131	2·131	576	*ms*
202	2·119	—	1	—
301	2·117	2·121	299	*m*
020	2·037	2·037	456	*m*
212	1·880	1·879	41	*w*
311	1·878		95	
121	1·836	—	2	—

Table 4 (*cont.*)

hkl	d_c	d_o	I_c	I_o
220	1·755	1·754	48	*vw*
302	1·748	—	4	—
103	1·732	1·734	< 1	*vvw*
400	1·728	1·724	2	*vvw*
221	1·668	1·668	109	*m*
401	1·645	1·646	290	*m*
013	1·638	—	30	—
022	1·622	—	35	—
312	1·606	1·605	257	*ms*
113	1·594	1·594	311	*ms*
410	1·591	1·590	133	*m*
203	1·589	—	18	—
122	1·579	1·580	492	*ms*
411	1·525	1·524	2	*vvw*
213	1·480	1·480	63	*w*
222	1·468	—	2	—
321	1·468	1·468	261	*ms*
402	1·453	1·452	< 1	*vw*
303	1·413	1·413	300	*ms*
412	1·368	—	10	—
004	1·342	1·341	177	*w*
501	1·339		65	
313	1·335	1·335	152	*w*
322	1·326	—	7	—
123	1·319	—	1	—
420	1·318	—	4	—
104	1·317	1·316	62	*w*
031	1·316		15	
131	1·293	1·293	154	*m*
421	1·280	1·279	630	*ms*
511	1·271	1·272	136	*m*
230	1·264	1·264	251	*m*
114	1·253	1·253	101	*w*
223	1·253		46	
204	1·251	—	50	—
403	1·243	1·242	85	*m*
231	1·230	1·230	453	*ms*
502	1·229	—	8	—
214	1·195	1·195	436	*s*
132	1·193	1·193	149	*ms*
413	1·188	1·188	5	*vw*
422	1·183	—	16	—
512	1·176	1·176	1043	*s*
323	1·161	1·161	488	*ms*
304	1·159	—	< 1	—
600	1·152	1·152	349	*w*

In the *B*27 structure, the atomic positional parameter y is fixed at 0·250 for both Lu and Ni. The atomic positional parameters x and z for Lu are assumed to be respectively 0·185 and 0·144. For Ni the x and z parameters are assumed to be 0·040 and 0·653. The good agreement between calculated and observed intensities indicates that the assumed values are not greatly in error.

The structure of TbNi could not be determined. An intensive effort was made to index powder patterns of a series of alloys ranging from $Tb_{54}Ni_{46}$ to $Tb_{48}Ni_{52}$. Metallographic examination showed that $Tb_{51}Ni_{49}$ was the closest to being single-phase, and that a TbNi compound does exist. The diffraction pattern is more complex than either of the neighboring phases GdNi (B_f) or DyNi (*B*27). Two ternary alloys were investigated, $GdTbNi_2$ and $DyTbNi_2$. The former was indexed as B_f, the latter as *B*27. Thus, our data indicate that Tb will partially substitute for Gd in GdNi and for Dy in DyNi, but that TbNi alone has some unknown crystal structure.

A b.c. cubic structure was found in equiatomic compounds in which the A partner was Sc, Y, Sm, Gd, Tb, Dy, Ho, Er, Tm or Lu, and the B partner was Rh. Equiatomic compounds of Ir with Ho, Er, Tm or Lu also have a b.c. cubic structure. Superlattice lines of the CsCl type were identified in the diffraction patterns of ScRh, TmRh and LuRh. It seems likely that the other b.c. cubic compounds listed in Table 1 also have the CsCl-type structure. This belief is supported by the observation that ScRh (Compton, 1958) and ScIr (Aldred, 1962) are known to be CsCl-type structures. Lattice parameters are given in Table 1. Earlier work by the present authors (Nevitt, 1963) gave slightly different values for ErRh and LuRh. New alloys were used in the present work. Differences in composition may account for the discrepancies. Calculated and observed d spacings and intensities are given in Table 5 for HoIr.

Table 5. *Observed and calculated values of d spacings and intensities of* HoIr

Wavelength of radiation: Cu $K\alpha_1 = 1{\cdot}54050$ Å

hkl	d_o	d_c	I_o	I_c
100	—	3·381		< 1
110	2·38	2·391	*vs*	1000
111	—	1·952		< 1
200	1·69	1·691	*m*	178
210	—	1·512		< 3
211	1·38	1·380	*s*	735
220	1·195	1·196	*m*	112
300	—	1·127		< 1
221		1·127		1
310	1·069	1·069	*ms*	166
311	—	1·019		2
222	0·977	0·976	*m*	50
320	—	0·938		1
321	0·904	0·904	*s*	336
400	0·845	0·845	*w*	52
410	—	0·820		2
322		0·820		4
411	0·797	0·797	*vs*	671
330		0·797		168
331		0·776		4

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Elastische und thermoelastische Konstanten von K_2SO_4, Rb_2SO_4, Cs_2SO_4, $(NH_4)_2SO_4$, Tl_2SO_4 und $K_2Mg_2(SO_4)_3$

VON S. HAUSSÜHL

Kristallographisches Institut der Universität Freiburg i.Br., Deutschland

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The elastic behaviour of the K_2SO_4 isotypic crystal group confirms the general validity of rules about the influence of lattice dimensions and properties of constituents on elastic and thermoelastic constants. Such rules were obtained earlier on isotypic cubic crystal groups. The elastic properties of $K_2Mg_2(SO_4)_3$ are strongly correlated with those of K_2SO_4.

Das Ziel der vorliegenden Untersuchung war es zu prüfen, ob die bisherigen Regeln über die Abhängigkeit elastischer und thermoelastischer Konstanten von den Eigenschaften der Bausteine in isotypen Kristallreihen auch bei Kristallen geringerer Symmetrie gelten. Die K_2SO_4-Gruppe weist in fast allen physikalischen Eigenschaften eine bemerkenswert geringe Anisotropie auf. Die Abhängigkeit der elastischen Eigenschaften von den Bausteinen wird daher nur unwesentlich vom Einfluss der Anisotropie verwischt, sodass hier die Möglichkeit zu einer direkten Anknüpfung an die Verhältnisse bei kubischen Kristallen besteht. Ein Vergleich des elastischen Verhaltens des kubisch kristallisierenden $K_2Mg_2(SO_4)_3$ mit K_2SO_4 wurde durch die freundliche Überlassung eines natürlichen Langbeinit-Kristalls durch Herrn Prof. Zemann, Göttingen, möglich, dem auch an dieser Stelle sehr herzlich dafür gedankt sei.

Herstellung der Kristalle

Alle Kristalle mit Ausnahme von $K_2Mg_2(SO_4)_3$ wurden aus wässrigen Lösungen durch Senken der Temperatur bzw. langsames Eindunsten des Lösungsmittels bei etwa 50°C gewonnen. Bei Tl_2SO_4 konnte das Wachstumsvermögen durch Zusatz von Schwefelsäure wesentlich verbessert werden. Die wasserklaren Kristalle erreichten Abmessungen von mehreren cm in allen Dimensionen. Es wurde folgende Rangfolge glatter Flächen (Aufstellnng nach Groth, 1908) beobachtet:

K_2SO_4:	{111), {011}, {021}, {110}, {112}, {130}, {100}, {001}, {010}.
Rb_2SO_4:	{111}, {021}, {010}, {011}, {112}, {001}, {100}, {130}.
Cs_2SO_4:	{010}, {001}, {111}, {110}, {021}, {011}.
$(NH_4)_2SO_4$:	{011}, {110}, {111}, {010}, {130}, {021}, {001}, {100}.
Tl_2SO_4:	{010}, {110}, {111}, {011}, {021}, {001}, {102}, {012}.

Kugelwachstumsversuche an K_2SO_4 ergaben nur geringfügige Änderungen gegenüber dem Wachstum grosser Kristalle (v. Engelhardt, Haussühl & Zinnow, 1962).

Messungen

Bei den rhombischen Sulfaten wurden die elastischen Konstanten aus den Ausbreitungsgeschwindigkeiten elastischer Wellen mit Hilfe des verbesserten Schaefer–Bergmann-Verfahrens (bei 15 MHz) in den rhombischen Hauptrichtungen [100], [010], [001] und in deren Winkelhalbierenden gemessen. Die Werte für 20°C sind in Tabelle 1 aufgeführt. Die Bezeichnung entspricht der Aufstellung von Groth (1908). Die thermoelastischen Konstanten für 0°C wurden aus der Verschiebung von Eigenfrequenzen dicker Platten mit der